

PROGRESS REPORT NO. 2
THE FEASIBILITY OF MEASURING
THE MOISTURE GRADIENTS IN
CONCRETE PAVEMENT SLABS

AUGUST, 1959
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Joint
Highway
Research
Project

PURDUE UNIVERSITY
LAFAYETTE INDIANA

by
J.R. BELL

Progress Report No. 2

THE FEASIBILITY OF MEASURING THE MOISTURE GRADIENTS
IN CONCRETE PAVEMENT SLABS

TO: K. B. Woods, Director
Joint Highway Research Project

FROM: H. L. Michael, Assistant Director
Joint Highway Research Project

August 5, 1959

File: 9-7-3
Project: C-36-63C

Attached is Progress Report No. 2 on the "Feasibility of Measuring the Moisture Gradients in Concrete Pavement Slabs." The report has been prepared by Mr. J. R. Bell, Research Assistant, under the direction of Professor G. A. Leonards.

This progress report is the final report on the preliminary investigation phase of this research project, a cooperative project with the State Highway Department of Indiana and the Bureau of Public Roads. Several recommendations for further study are made on the basis of the findings of this preliminary study that the dielectric constant is a satisfactory indicator of the water content of hardened concrete and that the capacitance method of measurement is worthy of an intensive study.

This report is also accompanied by a proposal to extend the cooperative study with the State Highway Department and the Bureau of Public Roads to include some of the recommendations of this report.

The report is submitted for the record.

Respectfully submitted,

H. L. Michael

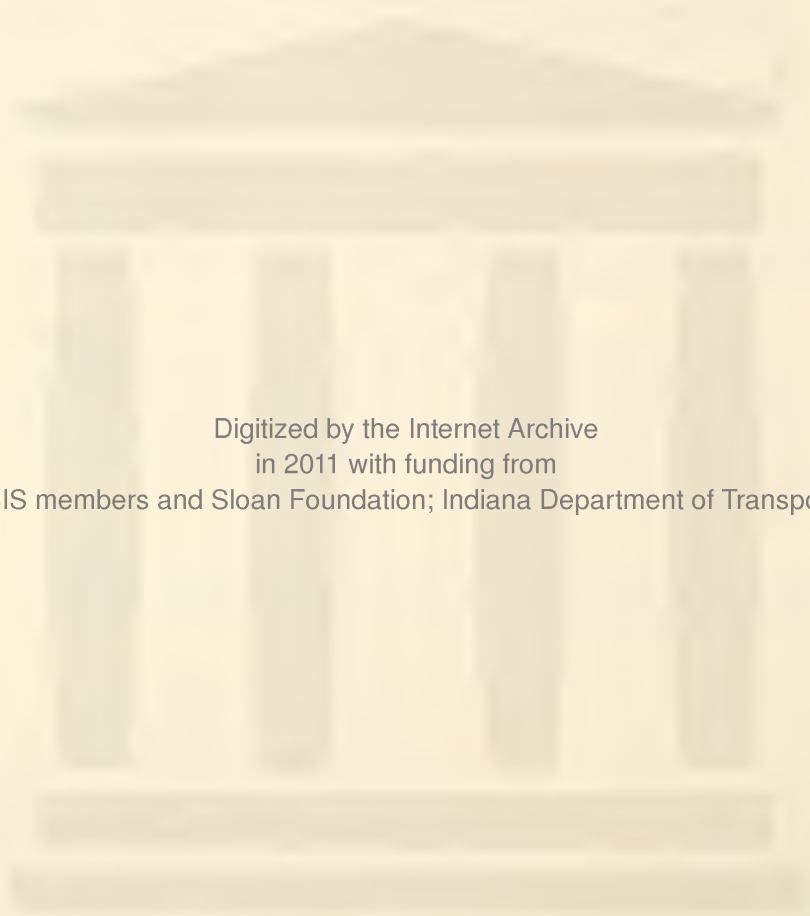
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Research Assistant

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Purdue University
Lafayette, Indiana
August 5, 1959

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SUMMARY

The purposes of this study were: 1) to conduct a comprehensive literature search to evaluate the available methods of measuring the moisture content of porous media, 2) to conduct limited laboratory investigation of the more promising of these methods, and 3) to select the most promising methods for further intensive investigation.

The study was divided into two parts: 1) a literature search and 2) a preliminary laboratory investigation of the dielectric constant (or capacitance) method, which on the basis of the literature survey appeared most promising.

The initial laboratory tests consisted, for the most part, of measuring the capacitance of a thin mortar disk at various water contents in a parallel plate capacitor by means of a low frequency AC capacitance bridge. The results showed these measurements to be very sensitive to changes in water content at low values of water content. However, in the higher water content range the method lacked sensitivity. Companion volume-water content tests indicate that significant volume changes occur in the range of water content above the sensitive ranges of these tests. Therefore, from the standpoint of obtaining an absolute measure of water content, or of developing an indirect procedure for measuring volume changes by capacitance measurements, the results showed an apparent lack of sensitivity at higher water contents.

The literature survey, and careful study of the theory of capacitance measurements, did not indicate any serious inherent limitations to the capacitance measurement method. From this standpoint, the basic method still appeared promising. It was concluded, therefore, that the difficulties encountered were the result of instrumentation problems.

For the most part, the instrumentation problems stemmed from the fact that the apparent dielectric constant as indicated by capacitance measurements is not a true constant. It is a function not only of the material being tested, but also of the test conditions. The two most important factors are boundary conditions and the electrical frequency at which the test instruments are operated. The most troublesome boundary condition is that of obtaining good contact between the capacitor plates and the dielectric material being tested. Poor contact lowers the sensitivity of the measurements. The problems associated with frequency of operation are more complex. The test frequency controls the effects of such factors as conductance, polarization, anomalous dispersion, and force fields acting on the water molecules. The influences of conductance and polarization are reduced as frequency is increased, but the other two factors tend to reduce the sensitivity at high frequencies. The problem, therefore, is to balance the physical size and shape of the capacitor and the frequency and sensitivity of the measuring instrument to the electrical properties of the dielectric - in this case, moist concrete.

To test the feasibility of designing apparatus of suitable range and sensitivity, a second series of capacitance tests was performed at a higher electrical frequency - 600,000 cycles per second. At this frequency the

working moisture content range was greatly increased without seriously reducing the sensitivity. The capacitance measurements are subject to changes in the structure of the concrete with time and reflect the sorption hysteresis in the wetting - drying cycle; however, the test results show that the inaccuracies from these causes are not prohibitive. These factors also limit the accuracy of nearly all other indirect moisture measuring methods. The results of the high frequency tests show that for a well-cured mortar the method will measure moisture contents with an accuracy of $\pm .5\%$ of dry weight for water contents between 1% and 6%. This range brackets the moisture contents that are of importance from the standpoint of moisture - volume relationships in concrete highway slabs. The range of sensitivity can probably be extended by increasing the operating frequency above 600,000 c.p.s.

From the results of this study, it is concluded that the dielectric constant is a satisfactory indicator of the water content of hardened concrete and that the capacitance method is worthy of an intensive study to develop the instrumentation necessary to permit routine insitu moisture measurements of hardened concrete.

INTRODUCTION

A knowledge of the moisture content of hardened portland cement concrete is of importance in a number of practical problems. Of particular interest to the present study is the effect of moisture gradients on the warping of concrete pavement slabs.

Experimental work has clearly demonstrated the significance of warping both on the development of stresses in the slab (1, 2)* and on the interpretation of pavement deflection data due to loads (3). Earlier theoretical studies (4, 5) appear to be of limited value, due to the assumption that the slab maintains contact with its support at all points and at all times. In a study conducted at Purdue University (6a, 6b), a new theory has been developed that allows for the possibility of a portion of the slab losing contact with its support. In the solution of the resulting equations, length changes due to both temperature and moisture gradients were combined by the use of an equivalent gradient and coefficient of expansion. Accordingly, while this theory appears promising, experimental verification of the computed deflections and stresses is of paramount importance. At the present time, such experimental studies are hampered by the lack of suitable instrumentation for measuring the moisture content, or more specifically, the moisture gradient in concrete slabs. Precision instruments for other necessary measurements - deflection, temperature gradient, strain, and the like - are currently available.

The water content of concrete is, by definition, referenced to the oven-dry condition of the concrete. Therefore, a direct gravimetric

* The numbers in parenthesis refer to the bibliography at the end of this paper.

determination of moisture gradients in concrete slabs is not feasible. Clearly, non-destructive tests are predicated on the measurement of some other characteristic (for example, relative humidity) that is a function of water content. To obtain the water content, the quantity measured must be correlated with the water content, as determined by destructive test methods. However, in the problem of measuring the differential length changes resulting from moisture migration, it may be that some other characteristic; such as, relative humidity or dielectric constant, is an equally good index of these length changes as the water content itself. If such is the case, this characteristic can be measured directly, and correlations with water content for the specific concrete mix can be eliminated.

If a non-destructive test method for measuring the moisture content (or its effects) in hardened concrete can be developed, the method can be applied to the solution of many other problems. For example, the investigation of stresses and deflections resulting from moisture gradients in structural units as well as in pavement slabs, and studies of moisture movements through walls and floor slabs. It is also possible that the method could be modified and applied to other porous materials such as soil, wood, and rock.

REVIEW OF LITERATURE

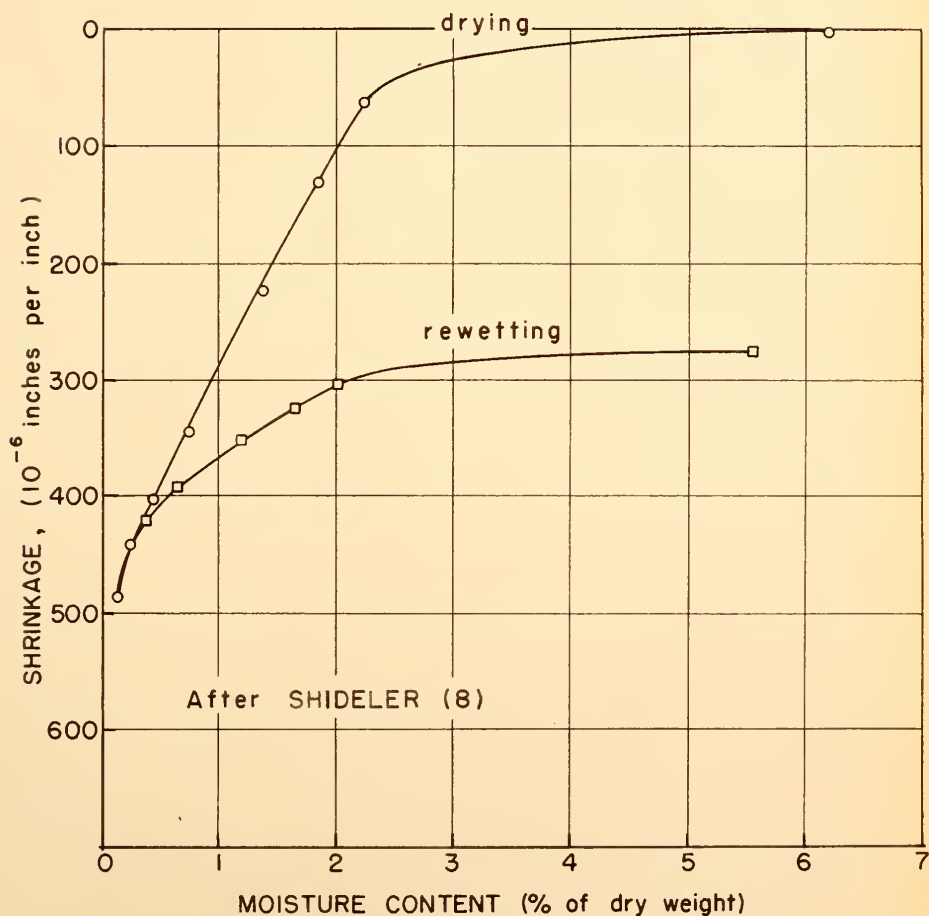
Moisture-Volume Relationships of Concrete

There is a great deal of literature on the water in concrete and its effect on the properties of concrete; however, much of the published information is qualitative rather than quantitative because reliable methods of measuring water content in non-destructive tests have not been available (1, 2). The published data are difficult to interpret because in many cases the descriptions of test conditions are incomplete. This has resulted in many apparent discrepancies; however, there are a few general trends, which are discussed below.

Menzel (7) states: "With all of the different types of concrete, whether rich or lean, dense or porous, etc., significant shrinkage begins at a moisture content corresponding roughly to the equilibrium value that would ultimately be attained with exposure to a 99 to 100 per cent relative humidity". Therefore, much of the water may be removed before appreciable shrinkage occurs. This limits the moisture content range of interest in this study to relatively low water contents. This statement is borne out by tests on masonry blocks by Shideler (8) and Kalousek (9). A plot of shrinkage vs. water content prep red from Shideler's data is shown on Figure 1. This curve shows that nearly all volume change occurred at water contents below 2 to 3 per cent by dry weight. This same general trend is shown by Yoshida (10) for cement paste; however, in the absence of aggregates, the shrinkage begins at somewhat higher water contents. See also the shrinkage data in this study (Figures 16-19).

The above data are not for concrete mixes used in highway pavements; however, they consistently show that nearly all shrinkage occurs at mois-

FIG.1.- SHRINKAGE vs. WATER CONTENT
FOR CONCRETE MASONRY UNITS.



ture contents lower than that corresponding to about 40 per cent of total absorption as determined by ASTM Designation C140-52. The results of tests by Blackman (11) show that total absorption for highway pavements would almost certainly correspond to water contents of less than 10 per cent by dry weight. This indicates that the critical moisture content range is below 4 per cent by dry weight. Teller and Sutherland (2) found by gravimetric tests the average moisture content of a slab at the Arlington Test Road to be 3.5 per cent in the summer and 3.8 per cent in the winter.

Theoretical considerations by Harr (6) indicate that it is desirable, with respect to slab warping studies, to measure the water content with sufficient accuracy to indicate length changes resulting from moisture variations equivalent to a temperature change of about 5°F. Hveem (12) has shown that changes in the moisture content of 30 oven-dried thin concrete disks, sawed from cores from pavements, which were soaked for seven days resulted (for 2/3 of the specimens) in length changes greater than those resulting from a 90°F. temperature increase. Also, a study of Shideler's data (Figure 1) shows that in the critical range a 2 per cent moisture increase results in a volume increase equivalent to a temperature increase of 30°F. or more.

Many factors influence the volume-water content relationship of concrete other than just water content. Some of these factors are age, curing, mix design, type and size of aggregate, cycles of wetting drying, carbonation, and others. (7, 8, 9, 10, 13).

Moisture Measuring Methods General

Literature on the subject of moisture measuring techniques is extensive and there are several general reviews and bibliographies available (14, 15, 16). Most of the methods have been applied to concrete, soils, and a variety of other materials with widely varying degrees of success. The literature is very confusing, as one author will report a method to be very reliable while another reports it as less than worthless. Therefore, in this discussion an attempt is made to evaluate the various methods on their relative inherent advantages and disadvantages and not solely on their reported reliabilities.

The definition of water content, as used throughout this paper, is the ratio of the weight of water lost upon drying to constant weight at 105°C to the weight of solids at this temperature. Direct determination of water content is unsatisfactory for the purposes of this study because it generally requires destruction of the concrete slab during testing. A possible modification would be to construct the slab with removable segments which would periodically be weighed, oven dried, weighed and returned to the slab. This would cause discontinuities in the slab, could provide avenues for the entrance of water, would not give a continuous record because a long time delay would be required before equilibrium was re-established after the segment was replaced, and the oven drying would alter the structure of the segment so that it would not remain the same as the rest of the slab.

Indirect methods measure some property of the concrete which is a function of water content and can be used for in-place, non-destructive measurements. These methods all present similar problems of range and

sensitivity, the necessity of calibration, the speed of response to moisture changes, the effective zone of influence, determination of just what water the device actually measures, the effect of changes other than moisture (i.e., changes in density, temperature, structure, and salt concentration), degree of contact between the sensing element and the material being measured, the requirement that some object or cavity to be placed in the concrete and, in some cases, the necessity for a hygroscopic material surrounding the sensing element which is supposedly in moisture equilibrium with the material being tested.

The more promising indirect methods are discussed in the following section.

Moisture Measuring Methods - Discussion of Methods

Resistance Method - In this method, the resistance of the material to the flow of an electric current is measured. An A. C. current must be used to prevent polarization in the vicinity of the electrodes. The electrical resistance is a function of both the structure of the solids and the composition and quantity of the pore water; therefore, it varies with changes in water content. The equipment and procedures required are simple but have the important disadvantages that the resistance is a function of the composition of the pore water. The resistance varies considerably with changes in salt concentration as well as changes in water content; therefore, variations in the measured resistance may not indicate corresponding changes in the water content. Spencer (17) reports that leaching may increase the resistance by 40 per cent in concrete.

Several moisture cells have been manufactured using this principle. In these cells the electrodes are embedded in some porous medium, usually plaster-of-paris (18) or wrapped in a mullin or filter-glass fabric (19, 20). In using these cells, the whole cell is embedded in the material for which the water content is desired. The cell absorbs water of varying amount depending on the water content of the surrounding material and the resistance of the cell is measured as an indication of the water content of the material in question. This requires that the resistance of the cell be calibrated with respect to the water content of the particular specimen being tested. These cells are subject to the same disadvantages stated above except that dissolved gypsum in the plaster-of-paris block acts as a buffer and reduces the effect of variations in salt concentrations in

the pore water of the material being tested. An additional problem is that there may be a time lag between the change in the water content of the concrete and the corresponding change of the moisture content of the hydrophilic material of the cell. This delay, if long enough, would be a serious problem when the moisture gradient must be measured under non-steady state conditions. Also the nature of the hygroscopic material limits the range of moisture content the cell can absorb and measure.

A recent modification of the resistance method worthy of special note is the Ionic Barrier Moisture Meter (21, 22). In this meter, the electrodes are surrounded by an ionic membrane which filters the ions from the water before it reaches the electrodes, thereby freeing the method from the disadvantage of being susceptible to changes in pore water composition. This method, in its present form, still has the disadvantage of measuring the resistance of a hydrophilic material (in this case the ionic membrane) instead of the concrete directly. Also, at this time the life of the ionic membranes is not known, but this modification seems deserving of special consideration.

Relative Humidity and Suction Methods - These methods measure factors which may prove to be better indices of the volume changes of concrete associated with moisture changes than water content per se (7). They can be related one to the other once and for all, and then, within certain limitations, related to water content for any given specimen (23). Relative humidity as used here is the ratio of vapor pressure at the surface of the water held in the porous material to the vapor pressure of a free water surface at the same temperature expressed as a percentage. Suction is the negative pressure (tension) of the water in the porous medium. Both are functions

of water content, structure, and to a limited extend composition of pore water.

The principal inherent problems associated with the use of these characteristics as measures of volume-water content relationships are: 1) the difficulty of finding one method that will measure these quantities over the full range of interest and 2) at high water contents, a relatively large change in water content results in only a very small change in relative humidity or suction. The advantage of using one of these quantities as an indicator of volume changes resulting from moisture changes would be that the equipment could be calibrated to read relative humidity (or suction directly). This would eliminate the necessity of calibrating the equipment for the specific specimen being studied. However, a relationship between humidity and the moisture coefficient of expansion of the concrete would still probably be required for the specific mix.

Relative humidity can be measured in a number of ways but the method which appears most logical for this study is the electric hygrometer method (24). The electric hygrometer measures relative humidity by measuring the electrical resistance of the air in the pores of the material. The resistance of the air depends on its water content. This may be measured either directly or indirectly by measuring the resistance of a hygroscopic material in equilibrium with the air.

Suction may be determined directly by measuring the force required to remove water from the porous material or indirectly by calculation from relative humidity. A vacuum method (25) is most frequently used for direct determinations but this method could not be used in this study because at the low water contents of interest the suction is greater than can be measured by vacuum methods.

The main advantage to these methods is that they are relatively free from effects of changes in pore water composition.

The problems of installing the hygrometers would be severe. They would have to be installed in cavities or they would simply be a modification of the resistance method and as such subject to all of the disadvantages of that method. The cavities themselves would be difficult to form and maintain without changing the moisture conditions in their immediate vicinity from that of the slab in general.

Radiation Methods - Radiations of all kinds through a porous material are affected by the presence of water in the pores. There are three kinds of radiation which might lend themselves to the purpose of this study; neutron, radio wave, and sonic radiations.

The neutron moderating moisture meter consists basically of a source of neutrons emitted into the material being tested and a detector which counts the number of slow neutrons reaching it. As the neutron moves through the material it is constantly colliding with the nuclei of the atoms in the material. Each collision slows the neutron. The magnitude of the reduction in velocity is a function of the relative sizes of neutron and the nucleus. The smaller the nucleus the slower the neutrons velocity after the collision. The hydrogen atom has a nucleus of mass equal to that of the neutron; therefore, the neutron is slowed much more by a collision with a hydrogen nucleus than by any other atom. Since almost all hydrogens in concrete are contained in the water the number of slow neutrons reaching the detector would be a function of water content. Neutron scattering indicates all water present-chemically combined water as well as evaporable water. This method has been studied by several investigators for moisture determination purposes (26)**

** Contains a very extensive bibliography.

The one probe neutron method which has the radiation source and the detector in the same probe probably could not be used because the volume of the zone of influence is too large to measure moisture gradients in thin slabs. If two probes, one with the neutron source and one with the detector, are used, the method is very sensitive to small changes in the distance between the probes unless the spacing is large. To control the size of the zone of influence the spacing would have to be quite small. Additional problems in maintaining the spacing of the probes would be encountered because, due to their high cost, the probes should be free to be moved from test point to test point. This presents the problem of forming and maintaining the cavities in the slab. Small, accurate spacing might make it necessary to embed the probes in the concrete, which would be very expensive. There are also the problems of designing a very small probe, danger in handling radioactive materials, and complex and expensive equipment.

The propagation of radio waves through a medium depends primarily upon the conductivity and dielectric constant of the material (27). Since both of these are functions of water content radio wave propagation is also a function of water content; however, this method simply combines all of the disadvantages of both the resistance and capacitance method without offering any special advantages.

The sonic method, which has been used to measure physical properties of concrete (i.e., Young's modulus, strength, etc.) (28, 29), measures the velocity of wave propagation through a material. The velocity of the wave disturbance is a function of the elasticity of the material which in turn is, among other things, influenced by water content; however, the precision required in measuring the velocity over the very

short distance necessary to establish moisture gradients in thin slabs would be very difficult if not impossible to obtain. Also, the changes in wave velocity resulting from factors other than moisture changes, such as deterioration of the concrete, would be much larger than the velocity variations from changing water content.

Heat Transfer Methods - Because of the high heat capacity of water, the heat transfer properties of porous materials are highly dependent on their water content. However, a serious problem confronting any effort to determine water content from measurement of heat properties is that, when a heat source is placed in a material such as concrete, thermal gradients are established which cause moisture migration thereby altering the moisture gradients as they are being measured (30). Further studies may show that these moisture movements occur so slowly in concrete that this problem may not be prohibitive. Aldous and Logton (31) tested more than 50 moisture cells of this type in connection with soils. Spencer (17) also discusses unsuccessful attempts to use this method with concrete. Another problem relates to the contact established with the material being tested and extreme sensitivity to minor variations in density and structure. There is also the problem of restricting the zone of influence to determine the gradient in a relatively thin slab. These cells, however, are relatively free from the influences of salts in the pore water (32).

Dielectric Constant or Capacitance Method - This method is very similar to the resistance method except that the dielectric constant of the material rather than its resistance is measured. Two plates forming a capacitor with the porous medium as the dielectric are embedded in the material to be tested. The dielectric constant of concrete varies with water content. The sensitivity of this method to slight changes in

moisture content may prove to be very high as the value of the dielectric constant of water is of the order of ten times that of mineral aggregates (33, 34). This method has an additional advantage in that the dielectric constant of water is only moderately susceptible to relatively large changes in salt content of the pore fluid.

Reference to the use of this method with concrete could not be found in the literature searched, but it has been used to measure the moisture content of soils (35, 36, 37, 38, 39), soybeans (40), wheat (41), cotton (42), gelatin and paper pulp (43), and others.

The apparent disadvantages to this method are mainly problems of instrumentation stemming from the fact that while the true dielectric constant of the material is a constant the apparent dielectric constant (the dielectric constant indicated by capacitance measurements) is a function of the frequency of the electrical current used to measure it. The problem, then, is to balance the physical size, shape, and spacing of capacitor plates and the electrical frequency of operation of the measuring instruments to the electrical and chemical properties of the concrete to give the desired sensitivity over the moisture range of interest. If all factors can be properly balanced, the size of the zone of influence can also be controlled.

The one apparent inherent disadvantage is that the dielectric constant of water is also a function of the force fields acting on the water molecules; therefore, the capacitance - water content relationship may show a hysteresis loop in the wetting and drying cycle and may also drift as the structure changes with continued hydration of the cement. It is, however, interesting to note that Andersen (35) found water content-capacitance curves of almost identical shape for five different granular

soils, indicating that possibly density and structure (except as it affects the force fields) may not have a large effect on dielectric constant of the water solids mixture.

SELECTION OF MOISTURE MEASURING METHOD

All of the indirect moisture measuring methods are to a greater or lesser extent subject to the same general problems and in addition some of the methods present special problems. In comparing the various methods, to select the most promising methods for laboratory testing, the following factors were considered:

1. Range and sensitivity
2. Calibration procedures
3. Response time
4. Size and shape of the zone of influence
5. What water is actually measured
6. The effect of factors other than amount of water present (temperature, density, structure, composition of water, and the like)
7. Dependence upon degree of contact between sensing element and porous material
8. Effect of installation upon moisture distribution in a concrete slab
9. Expense and complexity of equipment and operation
10. Special inherent problems
11. Special instrumentation problems

Pertinent moisture measuring methods and their more important advantages and disadvantages are outlined in Table 1. As most of the methods are subject to similar difficulties, a factor is listed only if it is relatively a serious disadvantage or an important advantage. For example, most of the methods are simple and inexpensive; therefore,

this item is not mentioned except in the case of the neutron radiation method where the comparatively complex and expensive equipment is a dead advantage, and in the case of the resistance method which is the simplest and least expensive method of all.

The resistance method was eliminated from consideration for laboratory tests because of its susceptibility to changes in pore water composition which may cause larger resistance variations than relatively large changes in water content.

The relative humidity and diffusion method, radiation methods, and heat transfer methods were rejected because while they offer no special advantages over the ionic barrier resistance method or the dielectric constant method they suffer from a wide variety of additional disadvantages. At first the ionic barrier method appears to be the most promising method; however, while this method is considered to be deserving of additional study, the capacitance method was judged superior for three reasons: 1) the ionic barrier requires a hygroscopic material surrounding the electrodes which increases its response time, 2) the useful life of the ionic barrier is unknown and 3) while the dependence of the measured capacitance upon the electrical frequency of measurement presents special instrumentation problems it also gives the capacitance method a degree of flexibility which none of the other methods possess. The factors which present problems at one frequency are not problems at some higher or lower frequency and it is believed that the proper combination of frequency and capacitor geometry will balance all of the factors involved to give a satisfactory sensitivity over the moisture range of interest and still provide freedom from the effects of small changes in

TABLE I - COMPARISON OF MOISTURE MEASURING METHODS

Method	Disadvantages	Advantages
Resistance Method	<ol style="list-style-type: none"> 1. Subject to variations in composition of pore water 2. Some methods require hygroscopic materials which increases response time and limits range 	<ol style="list-style-type: none"> 1. Very simple and inexpensive
Ionic Barrier Resistance Method	<ol style="list-style-type: none"> 1. Hygroscopic material required 2. Life of ionic membranes unknown 	<ol style="list-style-type: none"> 1. Free of effects of pore water composition. 2. Range of sensitivity may be controlled.
Relative Humidity and Suction Methods	<ol style="list-style-type: none"> 1. Range limited to very low water contents 2. Cavities required in concrete slab 3. Some methods require hygroscopic materials 	<ol style="list-style-type: none"> 1. Relatively free of effects of changes in pore water composition 2. Simple calibration procedure
Neutron Radiation Method	<ol style="list-style-type: none"> 1. Detects all water present--combined and evaporable water 2. Zone of influence large 3. Cavities required in concrete 4. Complex expensive equipment 5. Special instrumentation problems of designing special small probes and of maintaining critical probe spacing 	<ol style="list-style-type: none"> 1. Free of effects of changing pore water composition 2. Wide range 3. Relatively free of problems of contact between probes and material being tested

TABLE 1 (cont) - COMPARISON OF MOISTURE MEASURING METHODS

Method	Disadvantages	Advantages
R. F. Radiation Method	1. Combines disadvantages of both the resistance and the capacitance methods	3. No special advantages
Sonic Radiation Method	1. Extreme precision of measurement required 2. Very sensitive to changes in structure (deterioration of the concrete)	1. Free of effects of changing salt concentration of pore fluid
Heat Transfer Method	1. Serious problems of contact between sensing element and porous material 2. Sensitive to changes in structure and density 3. Measurements cause moisture migration 4. Large zone of influence	1. Free of effects of changing pore water composition
Dielectric Constant (capacitance) Method	1. What water measured - dielectric constant of water a function of the forces holding it in the concrete 2. Sensitive to degree of contact between the sensing elements and the material being tested 3. Special instrumentation problems - the measured dielectric constant is a function of the electrical frequency of the measuring instruments.	1. Relatively free from effects of small changes in composition of pore fluid 2. Rapid response to changes in water content. 3. Small zone of influence - well adapted to measurement of moisture gradients

composition of the pore fluid and the structure of the porous medium. This would permit the range, sensitivity, and zone of influence of the method to be varied to meet different requirements for different studies by varying the electrical frequency and capacitor geometry.

THEORY OF DIELECTRIC HYSTEROSCOPY

The following discussion of the dielectric constant is generally qualitative rather than quantitative because, while some of the factors influencing the dielectric constant can be expressed quantitatively, the interaction of all the contributing factors is so complex as to render the quantitative approach impractical. Therefore, only a general development of the theory will be attempted. The following discussions are based upon the works of MacLennan (44), Terman (45), Dörsey (33), and Harnsberry (46) except where otherwise noted.

The dielectric constant (ϵ) of a substance is defined by Coulomb's Law:

$$F = \frac{q_1 q_2}{Dr^2} \quad \text{-----} = 1$$

where F is the force exerted between two electrostatic charges q_1 and q_2 separated by a distance r in the substance. The dielectric constant of a vacuum is by definition equal to one; therefore, the dielectric constant of a material is equal to the ratio of the force (F_0) in a vacuum to force (F) in the material, all other factors being equal.

$$D = \frac{F_0}{F} \quad \text{-----} = 2$$

Dielectric constant is a dimensionless quantity with values equal to or greater than unity.

Substances composed of polar molecules possess high dielectric constants because in the presence of an electric field the polar molecules become oriented in such a manner as to oppose the field and, thereby, reduce the force in equation 2. Molecules may be distorted by the electric field. This displaces the centers of their positive and negative charges

and produces an "induced" dipole; therefore, non-polar substances may also have dielectric constants greater than unity. The dielectric constants of non-polar substances are, however, usually lower than the dielectric constants of polar materials.

Water molecules are very strongly polar giving water an abnormally high dielectric constant of about 80. The dielectric constants of most earth materials, which comprise the bulk of concrete, range from about 3 to 10. Therefore, the dielectric constant of a concrete is highly dependent upon its water content.

The dielectric constant of a material is a function not only of the magnitude of the polar moment (permanent and induced) of its molecules but also of the freedom of the molecules to be orientated by the applied electric field. If the molecules are already orientated and held by an existing field or are at a high energy level it will be more difficult for the applied field to orient them and the dielectric constant of the substance will be low. Therefore, the dielectric constant of a material is not truly a constant but is a function of temperature which influences the energy of the molecules (increasing temperature reduces the dielectric constant) and for polar substances such as water the dielectric constant is reduced in the vicinity of charged particles (i.e. ions and colloids).

Several equations have been proposed to permit the calculation of the dielectric constants of materials which are aggregates of substances with different dielectric constants. Most of the equations are based on models which do not truly represent the structure of concrete. The few methods which consider the structure of the material, for example the method developed by Brown (47), require experimentally determined shape

and/or distribution factors and are not applicable to this study. Bruggeman's equation (48), which assumes spheres in a matrix of different dielectric constant lends itself to the study of solid-water-air systems, and does not require prohibitively long calculations. This equation was modified by Cowrie and Palmer (37) to consider the effects of force fields acting on the water held very close to the spheres. They found good correlation between calculated values of dielectric constant and measured values for a clay. The solid curve in Figure 2a shows the general shape of the curve obtained by Cowrie and Palmer. They considered that the structure expanded as the water content increased and assumed saturation except at very low water contents. This is not the case with concrete. The theory was modified to consider a rigid structure of spheres consisting of a solid core with a water shell and air voids. The curve so obtained is represented by the dashed curve in Figure 2a. Neither of these models is considered to be sufficiently representative of the true structure to warrant quantitative consideration; however, the following theoretical values are given to show order of magnitude. At zero water content D is approximately 5 and for a water content of 20 percent (saturated) the calculated D is approximately 40 if force fields are neglected and about 15 if the effect of force fields as estimated by Cowrie and Palmer is considered. This last value appears to be too low. The work of other investigators (49) tends to show that the effect of force fields would be much smaller than that indicated by this value. The actual case for concrete is probably intermediate between the two curves.

If an electrical potential is applied between two conductors separated by a perfect insulating material an electrical charge is built up on the

FIG.2.- ILLUSTRATIONS OF DIELECTRIC CONSTANT THEORY

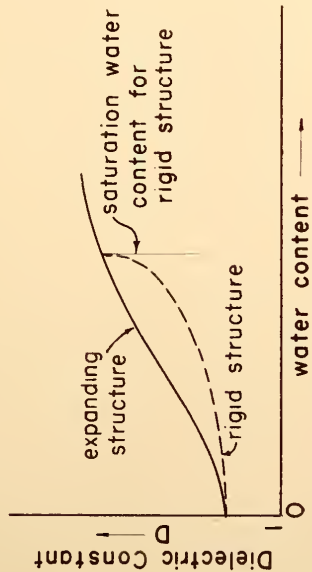


FIG.2a.-THEORETICAL DIELECTRIC CONSTANT vs. WATER CONTENT RELATIONSHIPS

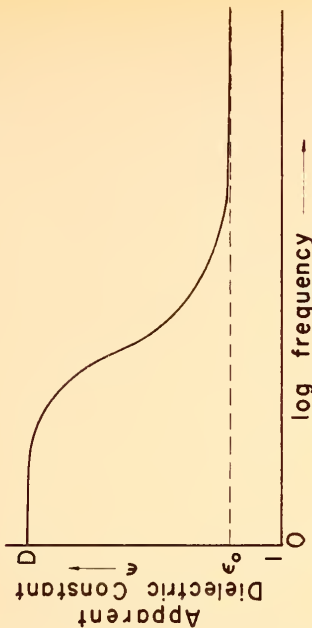
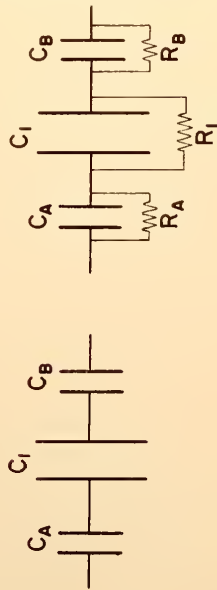


FIG.2b.- APPARENT DIELECTRIC CONSTANT vs. FREQUENCY FOR POLAR MATERIALS



(i) Perfect Dielectric (ii) Real Dielectric
FIG.2c.-EQUIVALENT CAPACITOR CIRCUITS

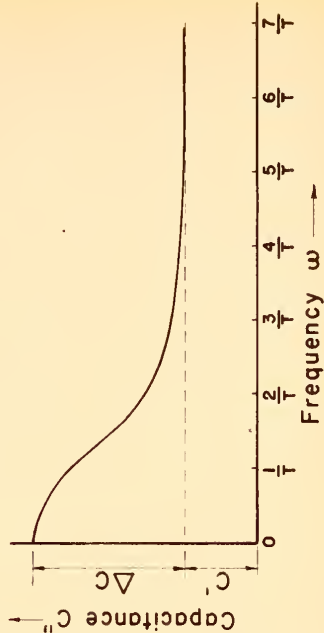


FIG.2d.- CAPACITANCE vs. FREQUENCY FOR REAL CAPACITORS

conductors. This arrangement of conductors (usually plates) and insulators is called a capacitor and the ratio of the charge to the applied potential is defined as the capacity or capacitance of the capacitor:

$$C = \frac{Q}{E} \text{ ----- } 3$$

where: C = Capacitance in farads (fd.)

Q = Charge on the capacitor plates in coulombs

E = Electric potential in volts

One farad is a very large capacitance; therefore, capacitance is usually expressed in $\mu\text{fd.}$ (10^{-6} farads) or Mfd. (10^{-12} farads).

The capacity of a capacitor is a constant dependent only on the size, spacing, and geometry of the plates and the dielectric constant of the insulating material. For parallel plate capacitors, the capacitance is expressed by the following formula.

$$C = K D \frac{A}{d} \text{ ----- } 4$$

where: A = area of the plates

d = distance between the plates

K = a constant selected to give capacitance in farads ($K = 8.84$

$\times 10^{-14}$ for A and d in square centimeters and centimeters

and $K = 22.45 \times 10^{-14}$ for values in square inches and inches).

From the above, it can be seen that the dielectric constant can also be expressed in terms of capacitance.

$$D = \frac{C}{C_0} \text{ ----- } 5$$

where C is the capacitance of a given capacitor with the material of dielectric constant D as the insulator and C_0 is the capacitance of the same capacitor with the dielectric replaced by a vacuum.

Dielectric constant is usually determined from capacitance measurements because of their simplicity. However, this introduces complications because although the dielectric constant of polar materials as defined for electrostatic fields is a constant for given temperature and force fields the apparent dielectric constant determined from capacitance measurements which must be made with alternating currents is not a constant but is a function of the frequency of the current used in the measurements. The symbol ϵ will be used throughout the remainder of this discussion to represent the apparent dielectric constant as indicated by capacitance measurements. The dependence of ϵ upon frequency results from the fact that with each cycle the induced electrical field reverses polarity and the dipole molecules, in order to remain oriented to the field, must rotate through 180° . The surrounding molecules offer a frictional resistance to this rotation and as the frequency increases all of the molecules are not able to change orientation as rapidly as the field reverses. For polar substances the plot of ϵ versus frequency assumes the general shape shown in Figure 2b. At very high frequencies the molecules are nearly stationary and maintain a random orientation so that the permanent dipole moment no longer contributes to the apparent dielectric constant. The induced dipole moment, however, is not interfered with by adjacent molecules and can keep up with the reversals at all frequencies; therefore, at very high frequencies ϵ approaches a constant value called the optical dielectric constant (ϵ_∞). Non-polar substances exhibit an essentially constant ϵ for all frequencies. This phenomenon is referred to as anomalous dispersion. The sharp break in the curve occurs at different frequencies for different substances. For liquid water the break comes at approximately 6×10^9 cycles per sec. and

for solid water (ice) at about 2.5×10^3 cycles per sec. The critical frequency for water very near charged surfaces is probably of the same order of magnitude as the value for ice (50).

The contacts between the capacitor plates and solid dielectrics are never perfect. If a space exists between the plate and the insulating material, the space has a capacitance. The capacitance developed at the contact between the dielectric and the capacitor plate is called the contact capacitance. To account for this contact capacitance a capacitor must be considered as three capacitors in series. This representation is illustrated by Figure 2e(1). In this figure, C_1 is the capacitance of the dielectric and C_A and C_B are the contact capacitances. If the contact capacitances are assumed equal, $C_A = C_B = \frac{1}{2}C_c$ where C_c is the total contact capacitance and the capacitor can be treated as two capacitors C_1 and C_c in series. For this condition,

$$C' = \frac{C_1 C_c}{C_1 + C_c} \quad \text{--- -- -- -- --} \quad 6$$

where C' is the total capacitance of a capacitor with imperfect contacts indicated by the capacitance measuring instrument. Equation 6 requires that the total capacitance must always be less than the smallest constituent capacitance; therefore, if the contact is very good making C_c very large compared to C_1 the value of C' will approach the value of C_1 and the effect of the contact capacitances can be neglected. On the other hand, if the contact is poor so that C_c is small the measured capacitance will approach a constant value equal to C_c regardless of the value of the dielectric constant of the dielectric. If the purpose of the capacitance measurements is to determine the dielectric constant of the insulator from the

values of C' , the method will be insensitive unless the contacts between the capacitor plates and the dielectric are very good.

The discussions thus far considered ideal capacitors incorporating dielectrics which are perfect insulators. Unfortunately, real dielectrics possess some conductivity, and this conductivity must be taken into account. Real capacitors can be considered as consisting of an ideal capacitor with a shunt resistance. The schematic representation of a real capacitor, considering both contact capacitances and conductivity, is shown in Figure 2c(ii). Assuming the contact capacitances and resistances to be equal $C_A = C_B = 2C_c$ and $R_A = R_B = \frac{R_c}{2}$ and the real capacitor can be represented by two capacitors in series: one with capacitance of C_c and resistance R_c representing the total contacts and the other with capacitance C_1 and resistance R_1 representing the dielectric. It is not possible to write a simple expression for total capacitance similar to equation 6 for this case because current passing through the capacitor is shifted so it is out of phase with the current passing through the resistance making the impedance of the network a complex variable with a real part and an imaginary part, which must be treated as vectors. However, by appropriate applications of the theories of alternating current electricity it is possible to derive the following expression for the total capacitance of a real capacitor (for derivation of equation 7 see Hartshorn (46)).

$$C'' = C' + \Delta C \text{ ----- 7}$$

where: C'' = total measured capacitance of a real capacitor

C' = geometric capacitance determined, for parallel plate capacitors, from equation 6 and 4 by substituting ϵ for D in equation 4

$$\Delta C = \frac{K C'}{1 + \omega^2 R^2 C^2}$$

$$\omega = 2 \pi f$$

f = electrical frequency in cycles per second

$$K = \frac{(R_1 C_1 - R_C C_C)^2}{C_1 C_C (R_1 + R_C)^2}$$

$$T = \frac{R_1 R_C (C_1 + C_C)}{R_1 + R_C}$$

From equation 7 the apparent dielectric constant as determined from capacitance measurements is clearly a function of the conductance of the material and of the frequency. A general plot of C' in terms of frequency is shown in Figure 2d.

As the water content of a porous material such as concrete is increased the apparent dielectric constant increases not only because of the increased ratio of water ($D \approx 80$) to concrete ($D \approx 5$) but also because as the water content increases the conductivity also increases. Therefore, even for a fixed frequency the measured dielectric constant of the water in a porous material is not a constant over the full range of moisture contents. Also, changes in the composition of the pore water will change the conductivity and the apparent dielectric constant. These effects are greatest for low frequencies and, as shown by Figure 2d, are insignificant for frequencies higher than about $4T^{-1}$.

In addition to influencing conductivity, the composition of the water affects the dielectric constant (D) of the water. First, as the salt content of the water is increased the force fields, from the ions in the solution, acting on the water molecules are increased and the dielectric constant is decreased. Data by Hasted, Ritson, and Collie (51) show that the effect of water composition is small except for very concentrated

salt solutions and indicate that the effect on anomalous dispersion is insignificant within the requirements of this study. Second and probably more important is the effect of electrode polarization. When a charged electrode, or capacitor plate, is placed in an electrolytic solution ions of charge opposite to that of the electrode are attracted to the electrode. This sheath of ions sets up a field opposing the field from the capacitor plates and causes an abnormally high value for the apparent dielectric constant of the solution. This problem has been studied by Smiley and Smith (52) and their data show that the apparent dielectric constant may be more than twice the true dielectric constant for low frequency measurements. In alternating current measurements the charge on the plate is constantly changing in sign and the ions are alternately attracted and repelled. At high frequencies there is insufficient time between alterations for the ions to move a significant distance and there is very little accumulation of ions on the plate. Smith-Rose (53) has shown that for frequencies above the low radio frequencies the effect of electrode polarization is very small.

There are several additional factors acting to produce discrepancies between the apparent dielectric constant indicated by capacitance measurements and the true dielectric constant which can not be evaluated. A few of these are heterogeneity of the dielectric, non-uniform moisture distributions, and fringe effects. Fringe effects are the effects of lines of force which do not extend directly across the space between the capacitor plates but loop outside of the zone between the plates. The effects of these factors should be relatively consistent for any given case and the resulting errors should be accountable by calibration.

The effect of all of the factors discussed above is to cause the ϵ - water content curves to deviate from the D - water content curves as shown in Figure 2a. It is impossible to predict what the net effect of all of these factors will be because very little is known about the dielectric properties of concrete or the concrete pore water. The actions and interactions of these several factors are complex but they do not appear to preclude a successful development of a capacitance moisture meter. The development of a successful capacitance moisture meter depends upon instrumentation which will satisfy three conditions. First, the frequency used must balance the effects of anomalous dispersion, force fields, conductivity, and electrode polarization, to give the steepest slope of the ϵ - water content curve in the moisture range of interest, and limit the effects of changing pore water composition. Possibly one frequency will be satisfactory for any given application or it may be desirable to use different frequencies for different moisture content ranges. Second, there must be good contact between the plates of the moisture indicating capacitor and the material being tested to insure consistent measurements and high sensitivity. If insulated capacitor plates are used the capacitance of the insulation must be constant with time and sufficiently high to provide adequate sensitivity at high water contents. Finally, the size and shape of the capacitor must be such as to have a large enough capacitance to permit accurate measurement and a zone of influence both large enough to give a representative water content and restricted enough to limit the measurements to a thin section of concrete so gradients can be determined. The problems involved are complex but a successful solution would provide an extremely flexible moisture measuring method whose range, sensitivity, and zone of influence could be varied to apply specifically to many different applications.

LABORATORY INVESTIGATIONS

The laboratory investigations consisted of two studies - 1) investigations of capacitance-water content relationships and 2) investigations of volume-water content relationships. The investigation of the capacitance-water content relationships was the primary study. In these tests the capacitance-water content curves for Portland cement mortar were determined at two electrical frequencies. Low frequency tests were performed at 60 c.p.s. and high frequency tests were performed at 600,000 c.p.s. The investigations of the volume-water content relationships were conducted to determine the required range of the capacitance moisture measuring method if it is to be satisfactory for use in volume-water content studies.

Test Procedures - 60 c.p.s.

The low frequency capacitance measurements were made with a 60 cycle General Radio Capacitance Test Bridge - Type 740 which measured both capacitance and power factor simultaneously. This bridge is similar to a D. C. Wheatstone Bridge except the power source is alternating current and the bridge is balanced by a variable capacitor and a variable resistance instead of only a variable resistance. The output of the bridge is fed into a tuned amplifier which in turn drives an electron-ray indicator tube. The variable capacitor and variable resistance in the bridge are adjusted simultaneously to balance the impedance of the unknown capacitance and null the bridge. A proper adjustment is indicated by a maximum spread on the indicator. The range of this bridge is $5/\mu\text{fd}$ to $100/\mu\text{fd}$ with a rated accuracy of $\pm 3/\mu\text{fd}$ in the range of measurements of interest in this study.

Preliminary Tests

The first tests attempted were capacitance measurements of a parallel wire capacitor, with the wires about 12 inches long spaced about $1/4$ inch apart embedded in two porous materials: plaster of paris and sand. These media were chosen for simplicity. The wires were insulated to keep the power factor within the range of the bridge. The parallel wire capacitor

in appearance closely resembled a conventional television antenna lead-in wire. This shape of capacitor was chosen because it would confine the zone of influence to a relatively thin section but would still give capacitances large enough to measure and would also average the moisture content over a fairly large volume. These tests showed significant capacitance changes with changes in moisture of the porous material; however, it was soon discovered that the insulation on the wire was absorbing water and changing the measured values. It was also apparent that the wire diameter and spacing and the insulation thickness were not suitable for optimum results. It was decided at this time that it was outside the scope of the study to develop the sensing element. Such tests were therefore abandoned.

A second series of tests was initiated to establish the capacitance-water content relationship of a Portland cement mortar. In these tests, the capacitance of a thin (about 4 mm) mortar disk 3 inches in diameter was measured at various water contents in a parallel plate capacitor (see Figure 3). The plates of the capacitor were insulated with Teflon (plastic) tape which has negligible water absorption.

The mortar disks were one part Type III cement to 2-1/2 parts of 16-100 sand mix at a water cement ratio of 0.65. The mortar was cast in a 3 x 6 inch cylinder cured 1 day in the mold and 3 days in a 100°F water bath. Upon removal from the bath the cylinder was sliced on a concrete saw and a disk was ground to the desired thickness and polished to give good contact with the capacitor plates.

After polishing the disk was vacuum saturated, weighed, and placed in the capacitor. The capacitor plates were adjusted for maximum capacitance reading (indicated best contact between plates and disk). The disk was left in the capacitor until an essentially constant value was obtained. This waiting period allowed the plastic insulation on the plates, which was not rigid, to adjust to the disk and was required to

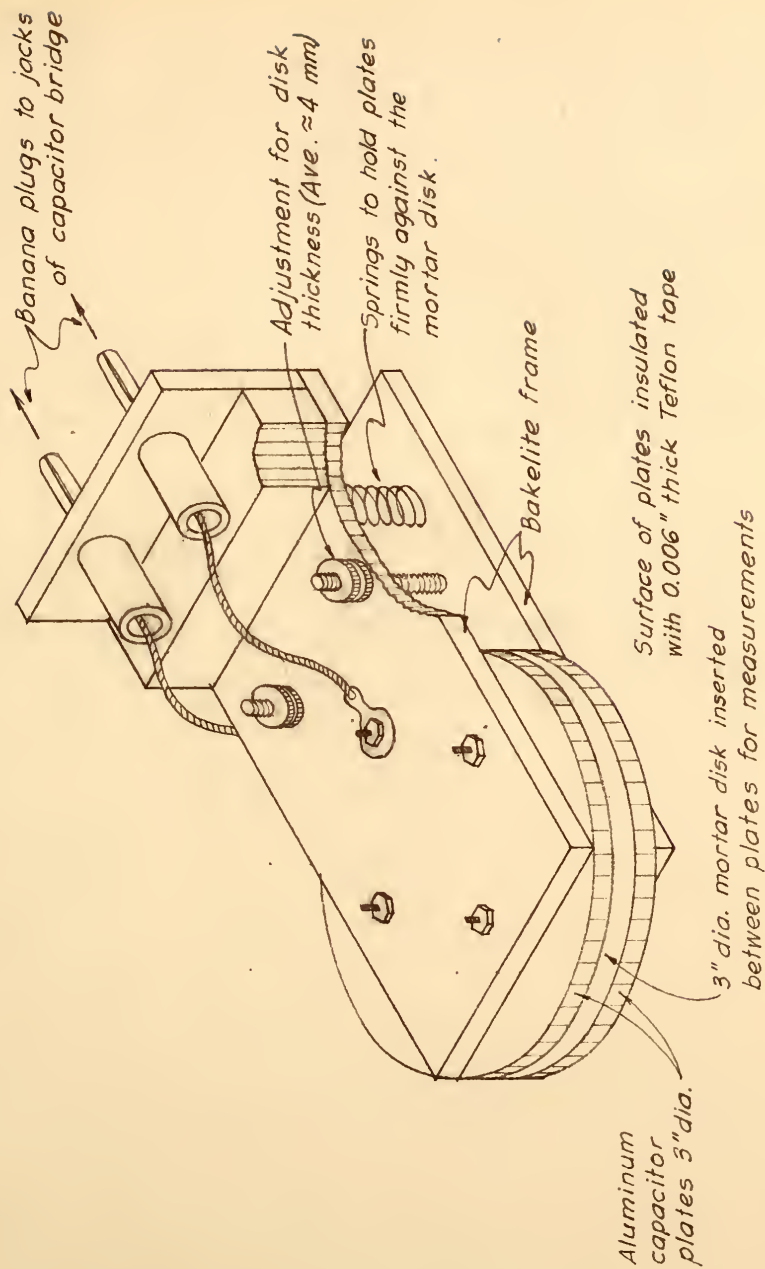


FIG. 3.- TEST CAPACITOR

Not to scale.

permit the moisture gradient through the disk to become essentially uniform. Subsequent experimentation showed that equilibrium was reached in about 1/2 hour. After the waiting period, the capacitance and power factor were recorded and the disk reweighed. All readings were obtained in this manner. The water content was lowered by drying in a vacuum desiccator. When the water content could no longer be lowered appreciably in a reasonable time the disk was dried 16 hours in the oven at 105°C. After drying the disk was cooled in a desiccator, weighed, and the capacitance was measured. All water contents were based on this dry weight. The water content was allowed to increase by absorbing water from the atmosphere and capacitance readings continued. When the disk would no longer absorb water from the laboratory atmosphere it was placed in a humid room and finally vacuum saturated in water.

The water content-capacitance curve obtained by this procedure had the same general shape as the curve shown for Disk #3 except the change in slope began at a slightly higher water content. On the second drying cycle it was observed that the curve had shifted to the right. This suggested some irreversible change upon drying, however, the dry weight at the end of the second cycle was higher than the first, and when the water contents for the second drying curve were recalculated using this higher dry weight the second curve nearly coincided with the first. This development made it impossible to determine whether there was a hysteresis loop in the curve, a general shifting of the curve due to structural changes, or just an apparent change due to increased hydration and carbonation during the period of the test; therefore, this test series was also discontinued.

Test Procedures Adopted

A third series of tests was designed to investigate the effects of continued hydration during the test and the resulting change in the structure of the mortar. For these tests, 15 disks were prepared as before with two exceptions: the disks were never allowed to become air dry before the tests and they were cured in 100°F water for 5 days instead of 3. After curing the disks were kept in water at room temperature except during grinding and polishing operations when they were kept surface wet. In addition, 5 disks were pressure steam cured for 5 days in an autoclave at a pressure of 150 psi.

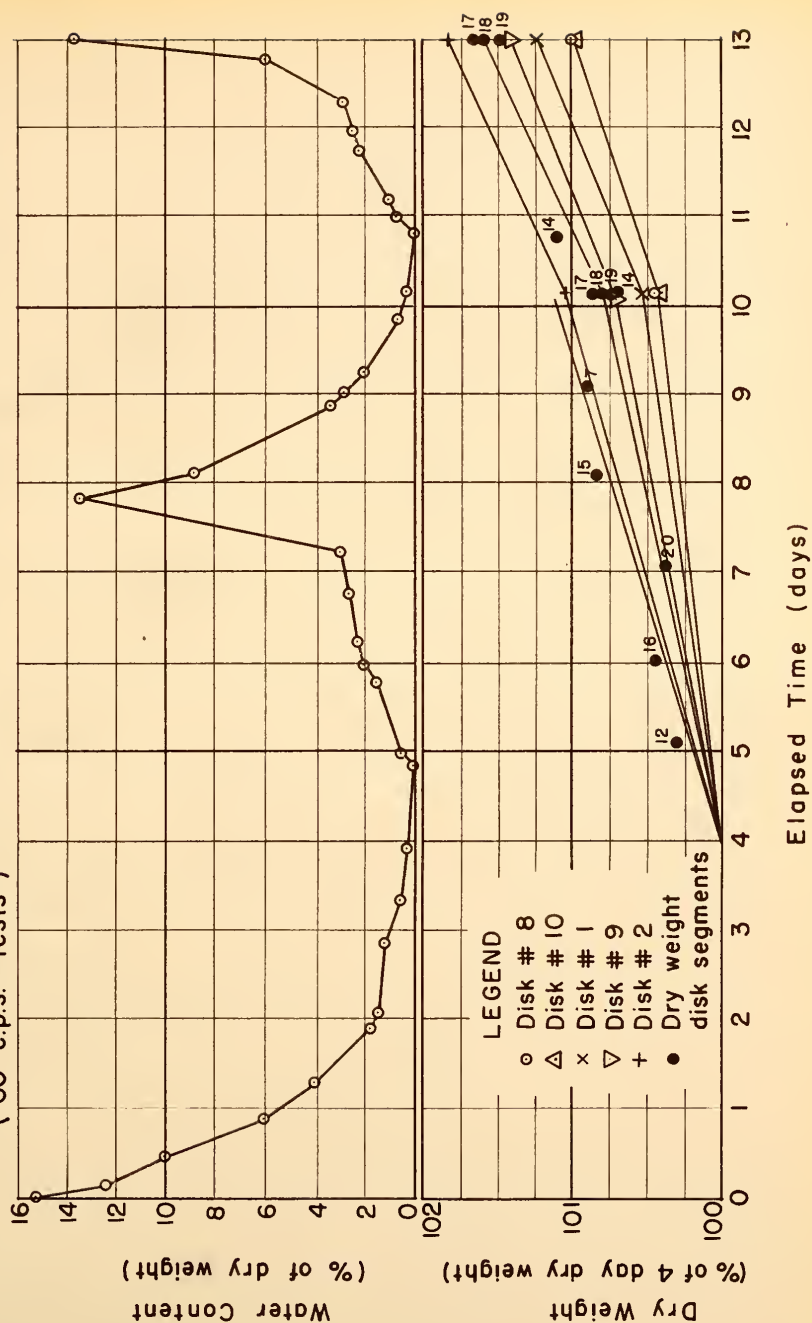
Capacitance measurements were made on three disks (2 hot water cured and 1 steam cured). The procedure was the same as that outlined above except that the edge of the capacitor was wrapped with a plastic ribbon during the waiting period of the capacitance measurement to maintain a more constant water content in the disk during this period.

Changes in the diameter of two other disks (one autoclaved and one hot water cured) were measured at approximately the time as the capacitance measurements were taken. All disks were treated, with respect to moisture environment, as nearly the same as possible throughout the testing program. The remaining 15 disks were periodically removed from the test one at a time, oven dried, and weighed. The purpose of this procedure was to establish the rate of increase of dry weight during the test so that the true water contents of the capacitance and shrinkage specimens could be computed at any time during the test. As these disks were wet at the beginning of the test the initial dry weight was calculated by breaking each disk, weighing both parts, and drying one segment

in the oven. The water content of the oven dry segment was calculated and, assuming that the initial water contents of both segments were the same, this water content with the wet weight of the other segment was used to calculate the initial dry weight of the wet segment. The dried segment was discarded and the moist segment was placed in the same moisture environment as the disks for capacitance and volume measurements until its time for oven drying. Later dry weights proved that evaporation during the time required for weighing of the wet segments caused appreciable errors in the calculated initial dry weight; therefore, the oven dry weight determined at the end of the first drying cycle was used as the dry weight without correction for the first drying cycle. The plot of the dry weight vs. time is shown on Figure 4. This figure also shows the moisture history of one of the capacitance disks during the testing period.

This third test series was continued through two cycles of wetting and drying.

FIG. 4 - DRY WEIGHT AND WATER CONTENT OF TEST DISKS vs. TIME
(60 c.p.s. tests)



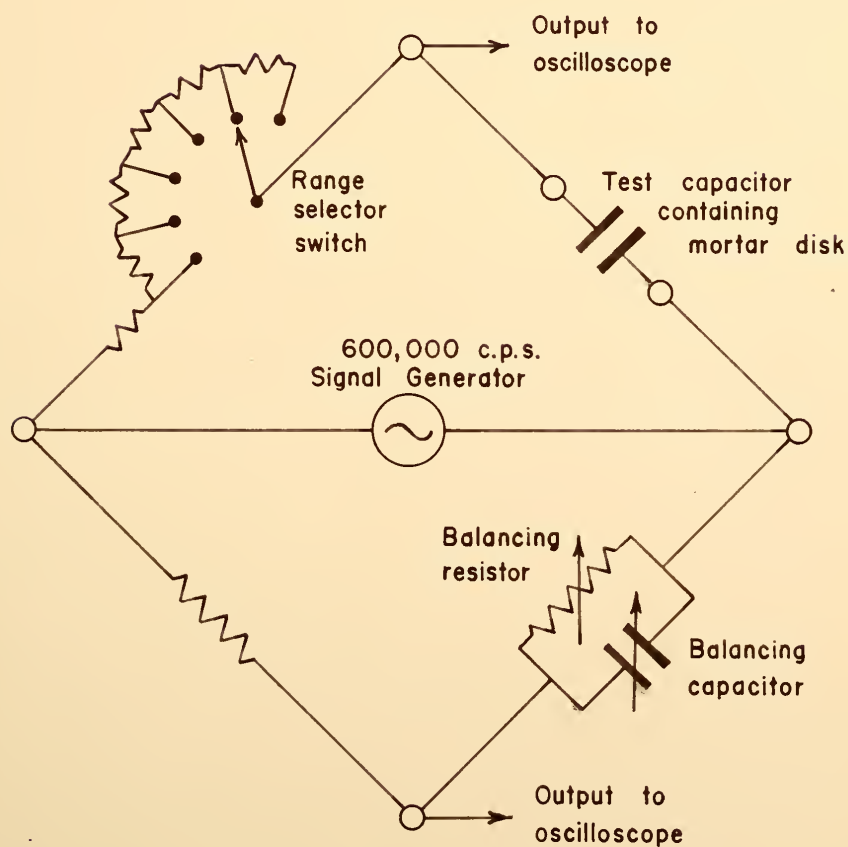
Test Procedures - 600,000 c.p.s.

The low frequency capacitance tests showed the method to be accurate at very low water contents but almost completely insensitive at higher water contents. Theory indicated that the range of sensitivity could be extended by increasing the electrical frequency of the capacitance bridge, and a series of capacitance measurements were initiated with a bridge operating at a frequency of 600,000 cycles per second. The test procedures followed in these tests were basically the same as those used in the low frequency tests except as noted below.

The most important change in the test procedure was, of course, the increase of the operating frequency from 60 to 600,000 c.p.s. A commercial high frequency capacitance bridge was not available; therefore, it was necessary to build a special bridge. The simplified schematic diagram of the 600,000 c.p.s. bridge is shown as Figure 5. The 600,000 c.p.s. input signal was obtained from a conventional commercial signal generator and an oscilloscope was used as an amplifier and null indicator. In operation the variable balancing capacitor and resistor were adjusted simultaneously to balance the effect of the test capacitor and null the bridge. Both readings were recorded. The bridge was not calibrated to give actual capacitance values for the test capacitor; therefore, the capacitance dial reading presented in the results is only a relative indicator of the actual capacitance of the test capacitor and mortar disk.

The full range of the bridge was divided into six segments by means of a range selector switch. This greatly increased the accuracy of the instrument by permitting the use of the full variation of the balancing capacitor for each segment or six times rather than just once for the full range of the bridge. The bridge was adjusted using Disk O (Figure 14). The full

FIG. 5 - SIMPLIFIED SCHEMATIC DIAGRAM OF 600,000
c.p.s. CAPACITANCE TEST BRIDGE



range of the bridge was not great enough to cover completely the capacitance variation of the disk; therefore, it was not possible to overlap all of the ranges and a gap was left between range five and six where measurements could not be taken. This gap in the measurements is indicated on plots of the results by the hatched zones in Figures 10 thru 14. The space left on the plots is not to scale with the rest of the data. In addition to these gaps in the data, variations in capacitance from disk to disk resulted in capacitance values at low water contents which for some disks were outside of the range of the bridge and, therefore, could not be measured. However, the main purpose of the high frequency tests was to investigate the capacitance-water content relationship at higher water contents where the records are complete.

A second important change from the procedures followed in the low frequency tests was in the sample preparation. In the preparation of the disks for the low frequency tests, extreme difficulty in polishing the disks was encountered because of the hardness of the quartz sand used in the mix. For the high frequency tests the same mix was used but a limestone sand was used instead of quartz. This made it possible to obtain a very smooth surface on the disks and the thicknesses of the disks could be controlled so that they were, for all practical purposes, of identical thickness. Also a Type I cement was used for these disks instead of a Type III.

Measurements were made on four disks in the high frequency test series. Three of these disks - A-C, B-C, C-C - were of the same mix (one part Type I cement to 2-1/2 parts 16-100 limestone sand at a water cement ratio of 0.65). Disks A-C and B-C were taken from the same cylinder which had been cured one day in the mold and five days in 100°F water and after slicing

had been stored in water at room temperature for 96 days before testing. Disk C-C had been given the same basic curing but had been stored for only one day before testing. Shrinkage measurements were made on three disks - A-S, B-S, C-S - as nearly identical to the above as possible.

The fourth capacitance test disk was Disk 8. This was one of the disks used in the low frequency capacitance tests. This disk was included in the high frequency tests for two reasons. First, it was considered advisable to have one disk made with the quartz sand and Type III cement for comparison purposes. Second, this disk had been subjected to several cycles of wetting and drying, had been oven dried several times, and had been allowed to air dry for several months so that it would be relatively stable and would not be subject to important structural changes during the period of the test. A comparison of the several test disks is presented as Table 2.

All disks were treated, with respect to moisture environment, as nearly the same as possible throughout the testing program except that Disks B-C and B-S were not oven dried until all tests had been completed. During the testing program these two disks were dried at a maximum temperature of 60°C. A record of the water content of the capacitance test disks versus time during the test period is given as Figure 6.

The greater uniformity in disk thickness eliminated the need for adjusting the spacing of the plates of the test capacitor for each disk. Therefore, the capacitor was adjusted once at the beginning of the testing program and this setting was maintained throughout the program.

In these tests, it was not necessary to provide a waiting period after the disk was placed in the capacitor for equilibrium to be established.

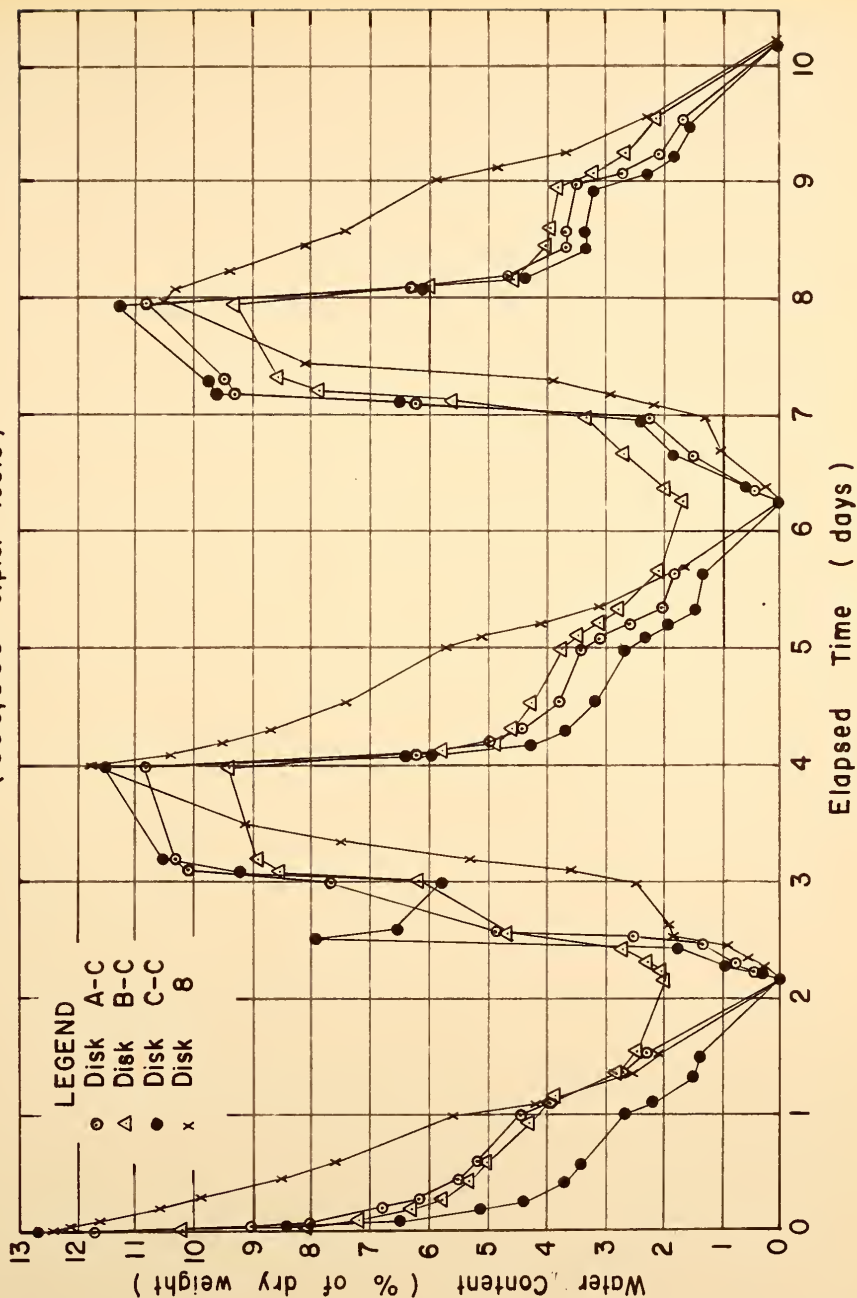
Table 2 - COMPARISON OF TEST CONDITIONS FOR MORTAR DISKS

Disk No.	Mix	Curing	Storage* Before Tests	Max. Drying Temp. During Tests	Test Fre- quency
8,9,10	1 part Type III Cement; 2½ parts Quartz sand (16-100); 0.65 water-cement ratio	1 day in mold 5 days in 100°F water	7 days in water at room temperature	105°C	60 c.p.s.
1,2	1 part Type III Cement; 2½ parts Quartz sand (16-100); 0.65 water-cement ratio	1 day in mold 5 days in 100°F water 5 days in autoclave (150 psi)	2 days in water at room temperature	"	
0	1 part Type I Cement; 2½ parts Limestone Sand (16-100); 0.65 Water-cement ratio	1 day in mold 5 days in 100°F water	76 days in water at room temperature	"	
A-C, A-S	"	"	96 days in water at room temperature	"	6×10^5 c.p.s.
B-C, B-S	"	"	"	60°C	
C-C, C-S	"	"	1 day in water at room temperature	105°C	
B	See B above	"	21.2 days, variable**	"	

* The disks were not allowed to air dry before the tests except for Disk #8 in the High Frequency test series.

** During this period this disk was used in the early low frequency tests, was allowed to air dry for several months, and was used in the first high frequency calibration tests. Exact records are not available but this disk had been subjected to several cycles of wetting and drying and had been oven dried several times before the start of the high frequency test series.

FIG. 6 - WATER CONTENT OF CAPACITANCE TEST DISKS VS TIME
(600,000 c.p.s. tests)



There was no change in capacitor dial reading with time. This was partially due to the reduction of electrode polarization at the higher frequency and partially due to a more stable condition of the plastic facing on the capacitor plates as a result of aging.

From the results of the hydration study conducted in conjunction with the low frequency tests it was concluded that such a special study was not warranted and the dry weights of the disks were adjusted for continuing hydration by simply pro-rating the increase in weight from one drying to the next over the intervening time.

The 600,000 c.p.s. test series was carried through two complete wetting-drying cycles plus an additional drying phase.

Test Results

The results of the low frequency capacitance - water content tests are shown in Figures 7 through 9. The capacitance values plotted on these figures are dial reading values for the total capacitance. These values include the contact capacitance as well as the capacitance of the mortar disks.

The results of the high frequency capacitance - water content tests are shown as Figures 10 through 13. The calibration curves for Disk O (Figure 14) are included here (even though this disk was not actually a part of the test series) for two reasons. First, it is the only set of data which shows the shape of the curve at very low water contents. Second, after two cycles of wetting and drying this disk was saturated with a 0.5 normal CaCl_2 solution and a third cycle was run using this solution. All other tests were performed using distilled water. The capacitance dial reading values plotted on these figures are not actual capacitance values but simply dial readings. These readings are a function of the capacitance of the test capacitor and mortar disk but the exact capacitance is not known; therefore, these values cannot be compared directly with the values from the low frequency tests. Figure 15 compares the capacitance dial reading vs. water content curves of Disks A-C and C-C after $1\frac{1}{2}$ cycles of drying and wetting.

The disks used in the tests were not identical. There were differences in composition, curing, and in the treatment of the disks before and during testing. These factors are important in the interpretation of the data, and, the special factors to be considered for each disk are indicated by a note on each figure. Each note states

FIG. 7 - CAPACITANCE vs. WATER CONTENT - DISK # 8
(60 c.p.s.)

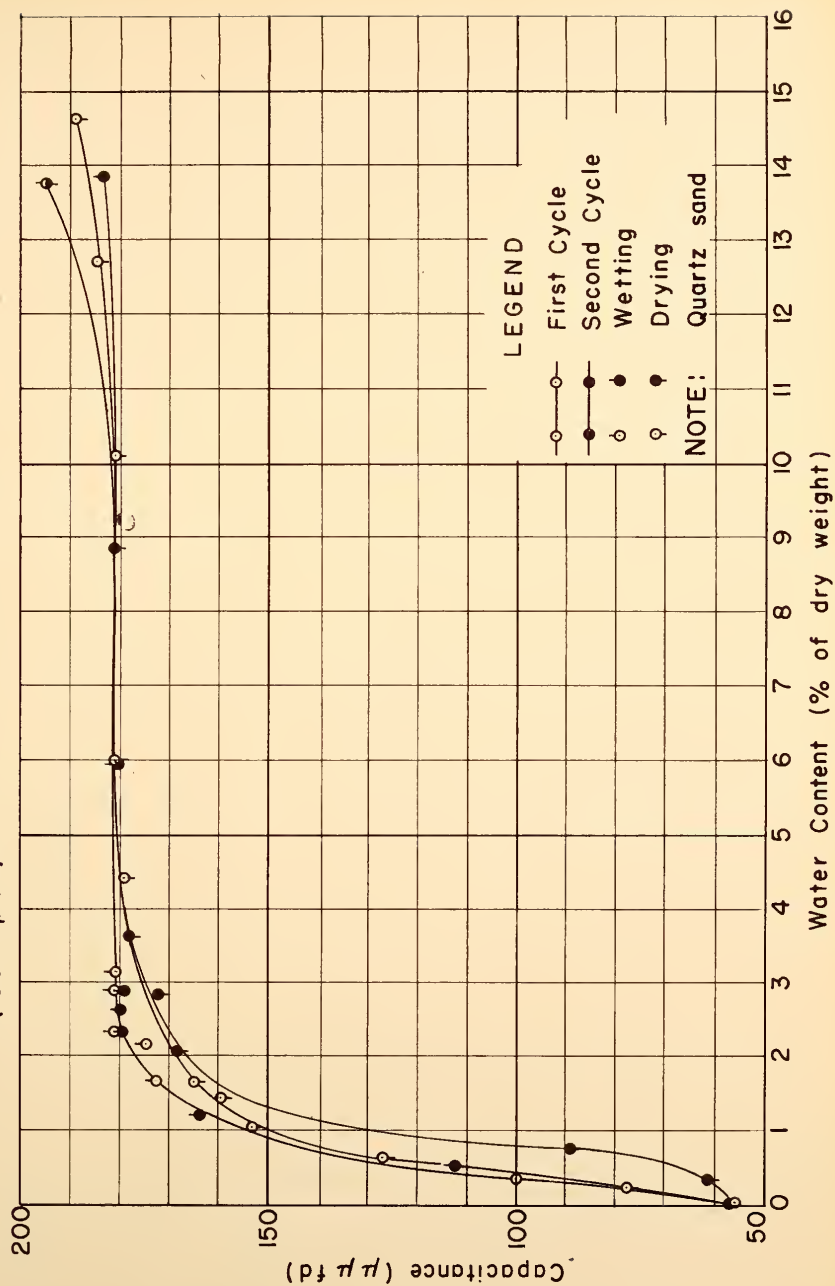


FIG. 8. - CAPACITANCE vs. WATER CONTENT.- DISK #10
(60 c.p.s.)

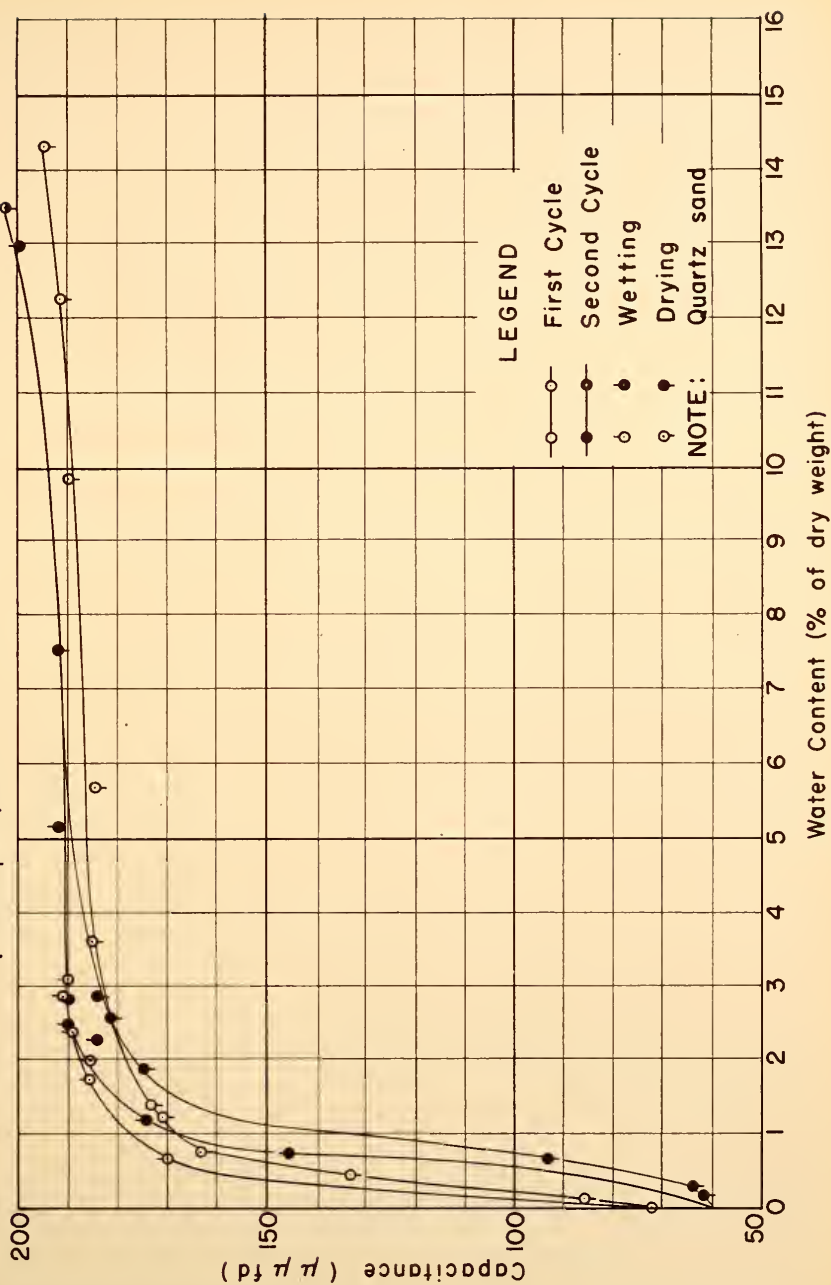


FIG. 9.- CAPACITANCE vs. WATER CONTENT.- DISK # 1
(60 c.p.s.)

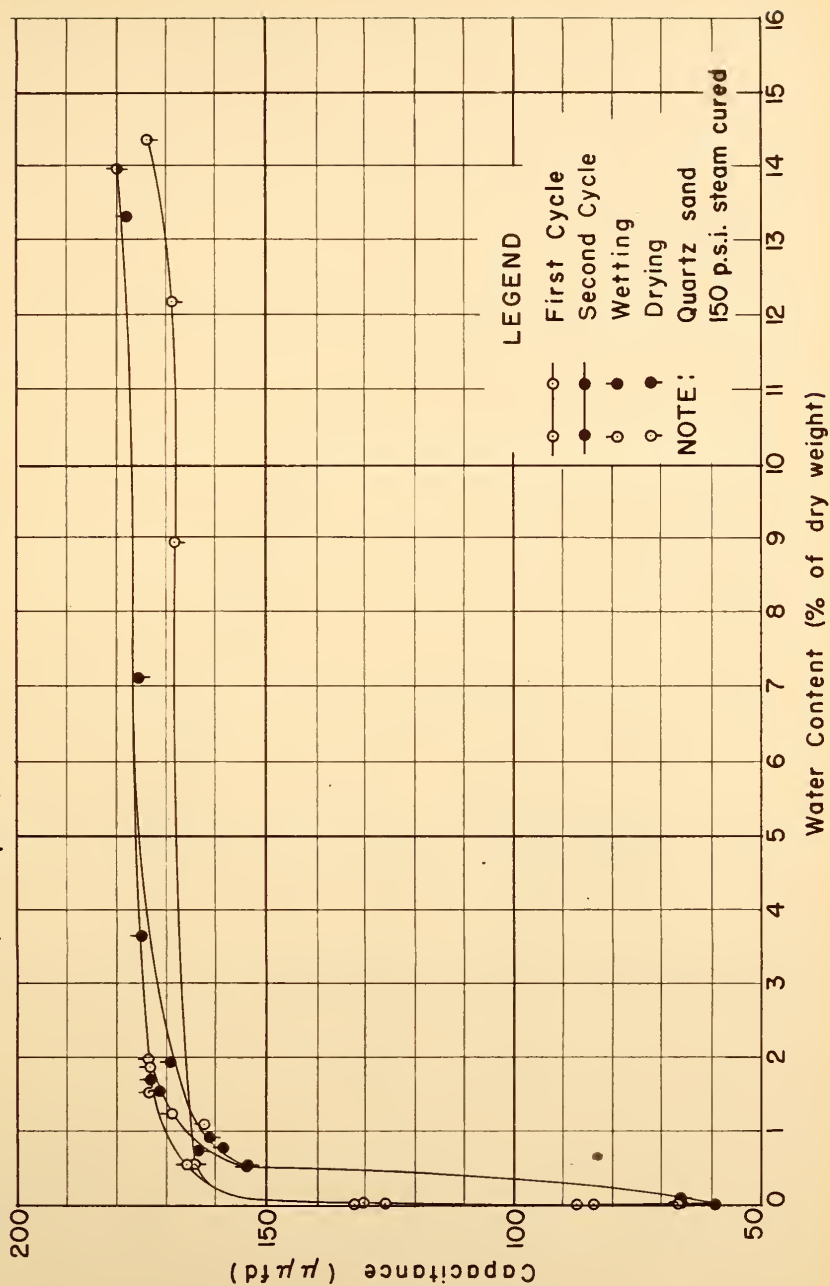


FIG. 10 -CAPACITANCE DIAL READING Vs WATER CONTENT-
DISK A-C (600,000 c.p.s.)

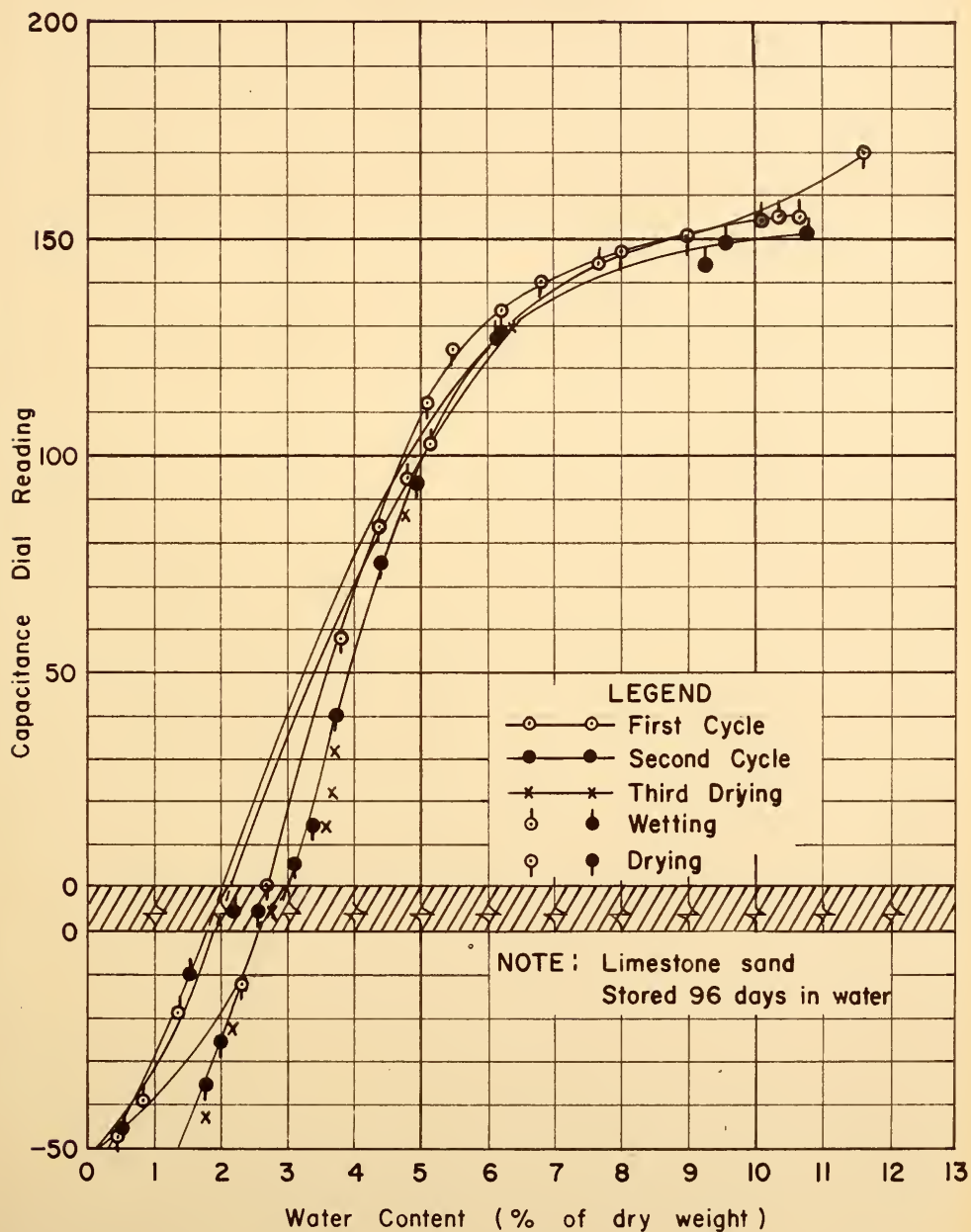


FIG. II - CAPACITANCE DIAL READING Vs WATER CONTENT -
DISK C-C (600,000 c.p.s.)

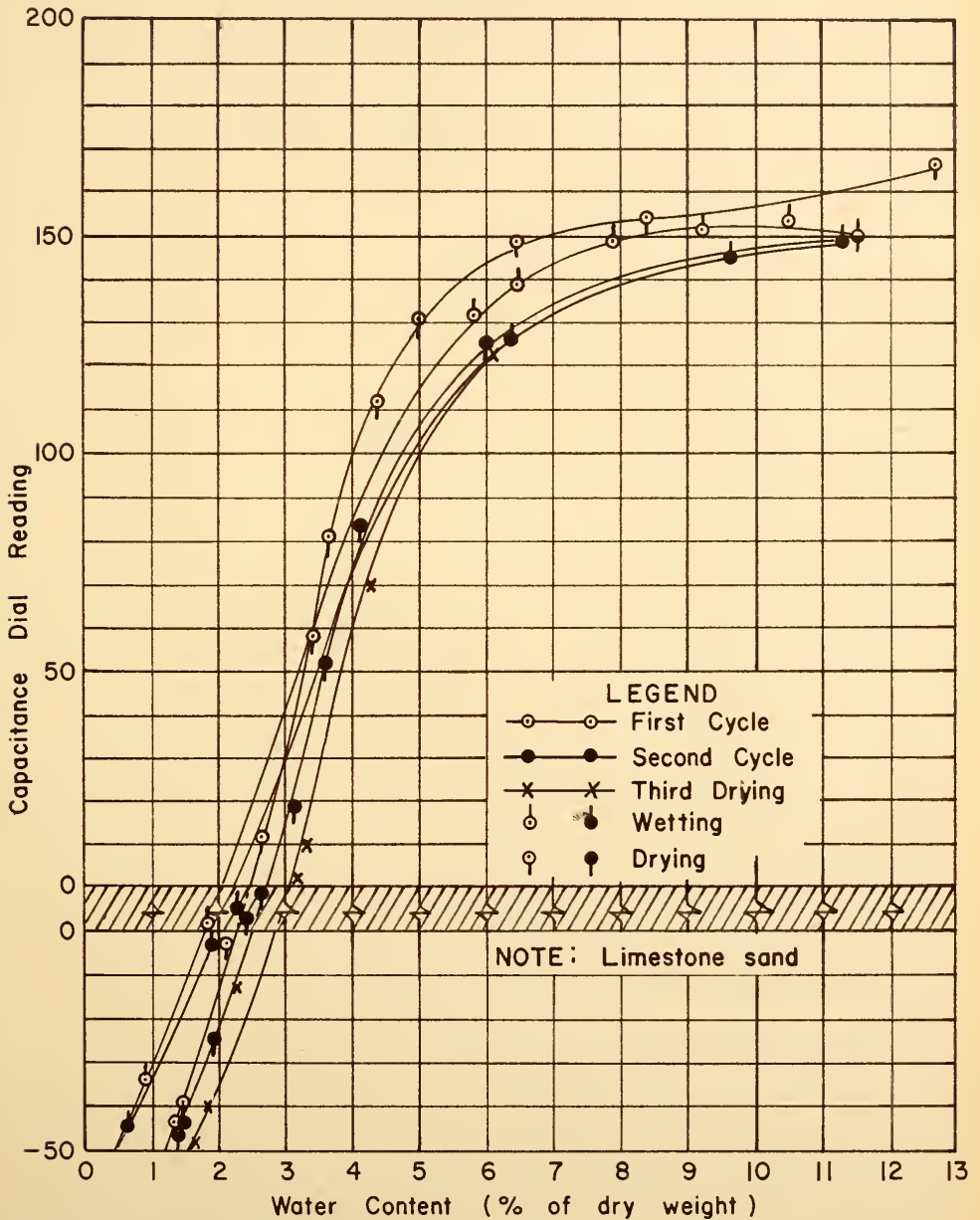


FIG. 12 -CAPACITANCE DIAL READING Vs WATER CONTENT-
DISK B-C (600,000 c.p.s.)

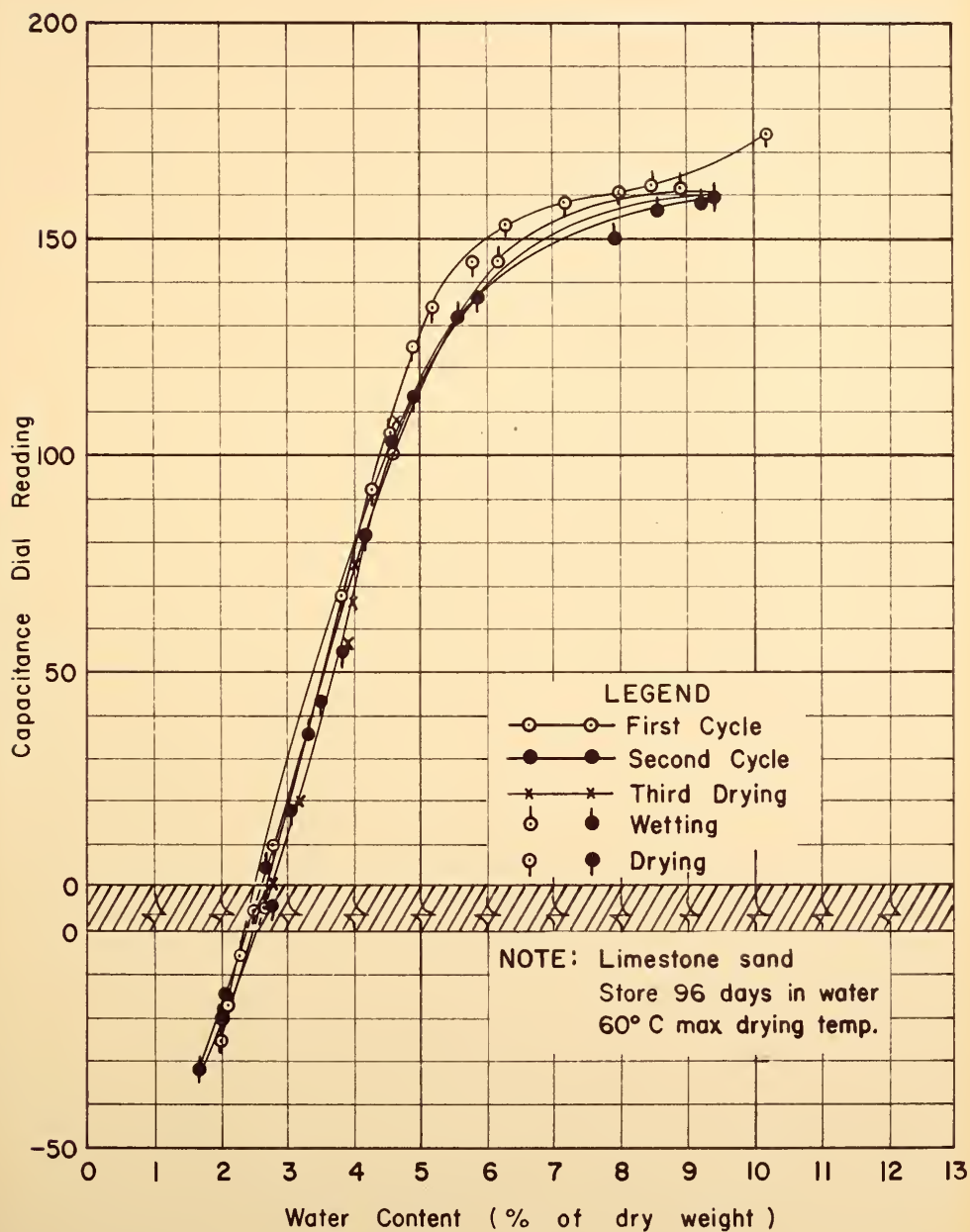


FIG. 13 -CAPACITANCE DIAL READING Vs WATER CONTENT-
DISK 8 (600,000 c.p.s.)

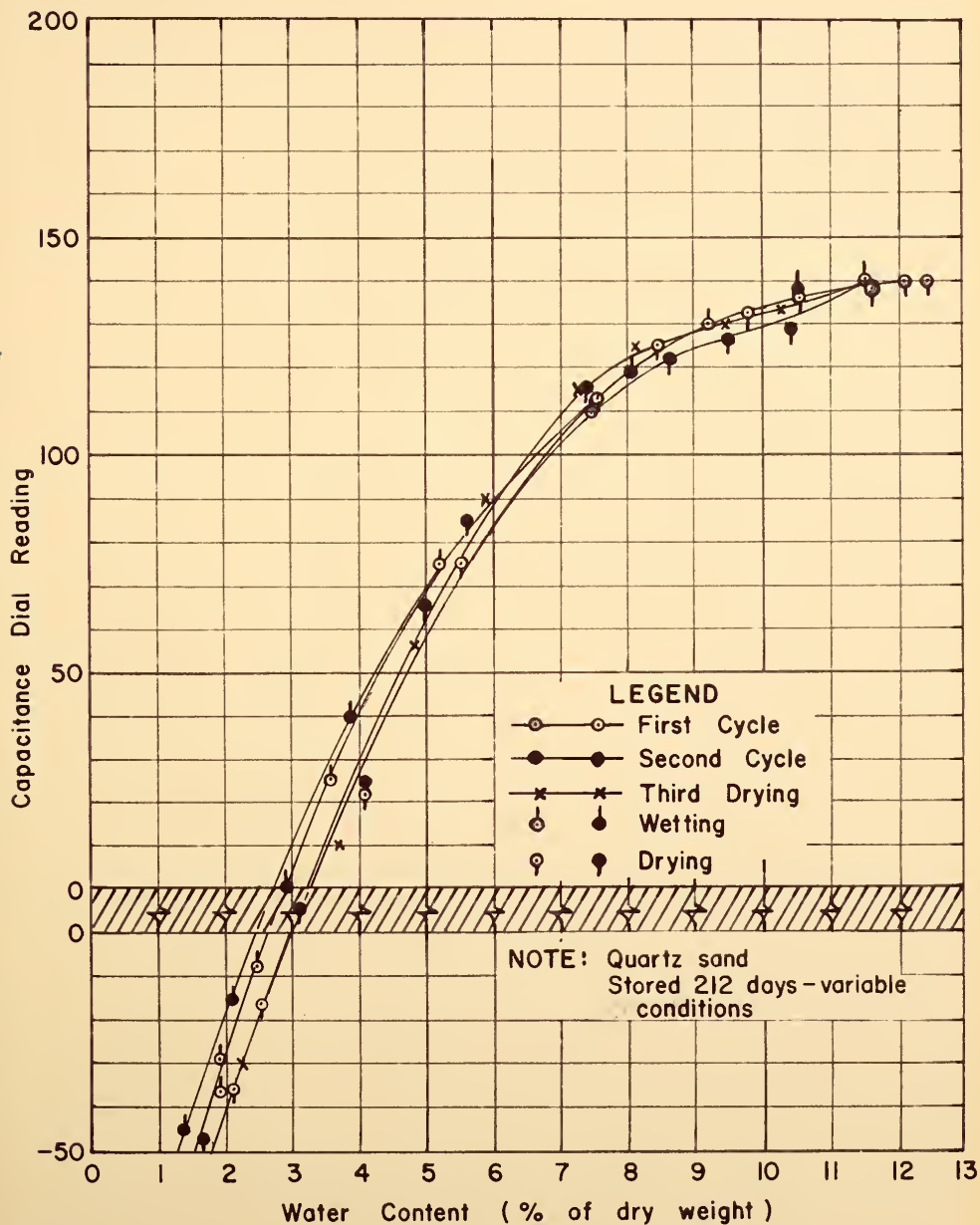


FIG. 14 -CAPACITANCE DIAL READING Vs WATER CONTENT-
DISK O (600,000 c.p.s.)

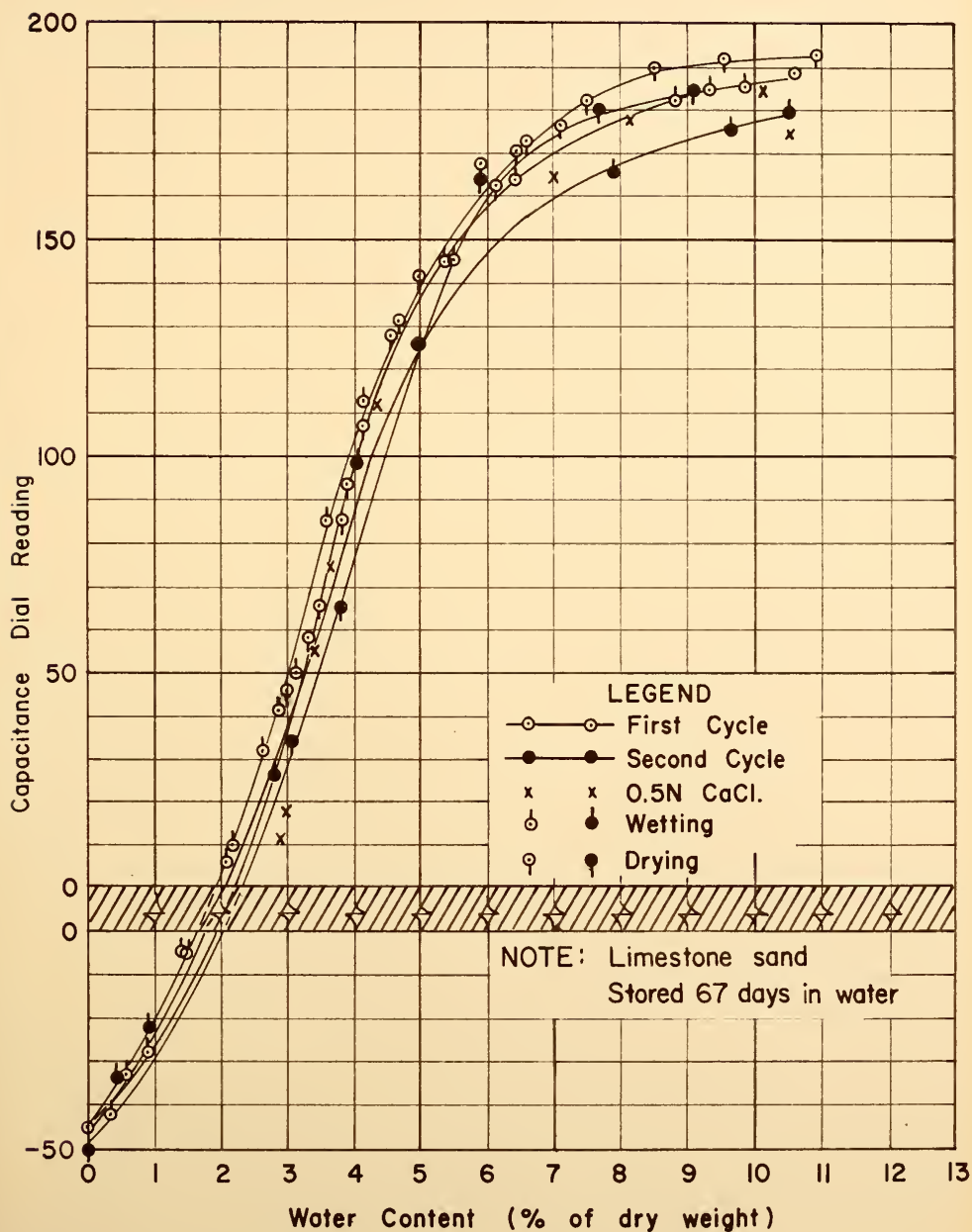
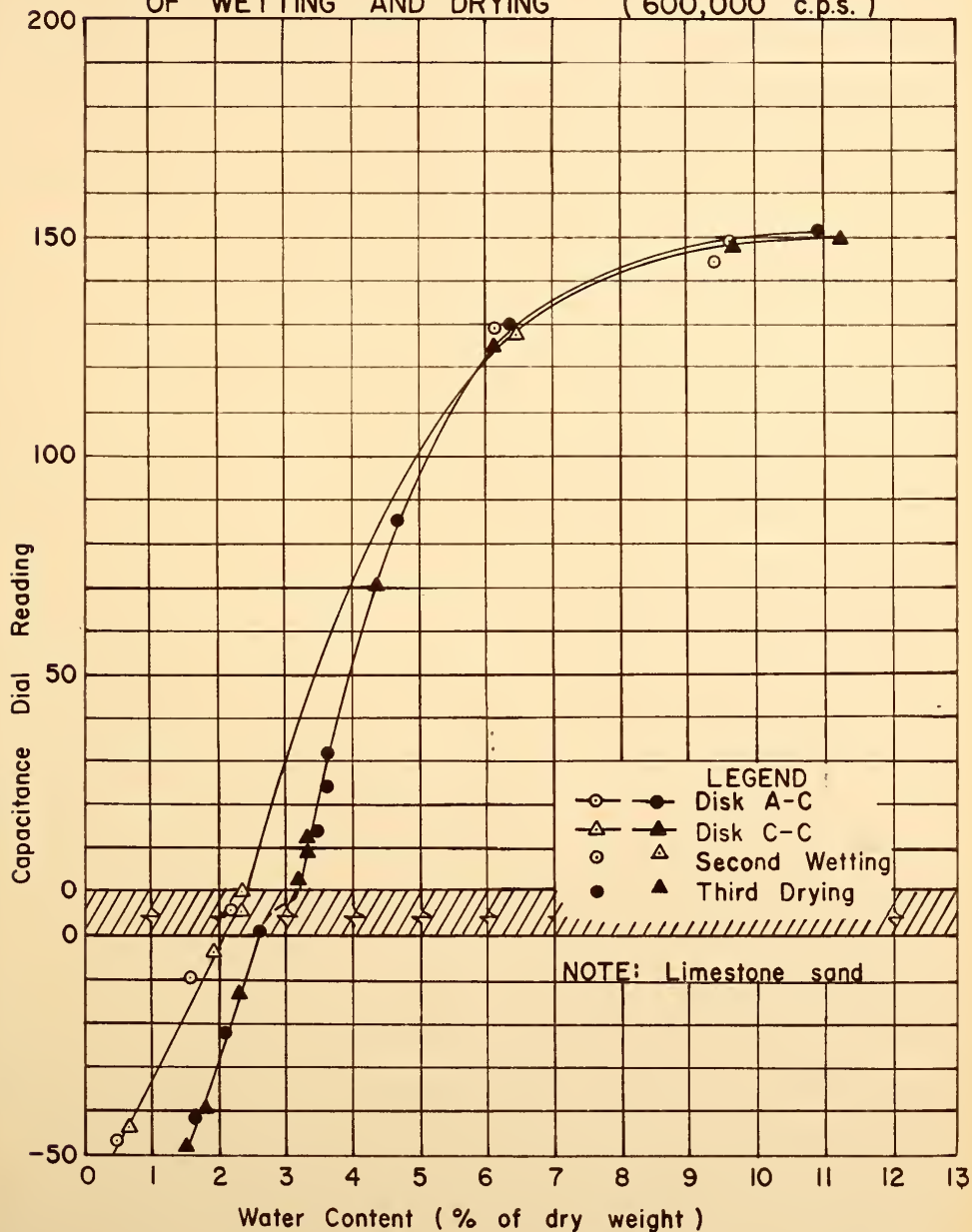


FIG. 15 - COMPARISON OF CAPACITANCE VS WATER CONTENT CURVES FOR DISKS A-C AND C-C AFTER $1\frac{1}{2}$ CYCLES OF WETTING AND DRYING (600,000 c.p.s.)



the type of sand used in the mortar (limestone or quartz) and the way the treatment of disk deviated from the standard. Standard treatment was a curing period of one day in the mold and five days in 100°F water, storage in water at room temperature for not more than one week before testing, and a maximum drying temperature of 105°C during the testing period. For a convenient comparison of the test disks see Table 2.

The results of the shrinkage-water content tests are presented in Figures 16 through 19. The shrinkage data are not presented for Disk A-S (companion disk to capacitance Disk A-C) because the brass plugs set in the disk to facilitate the measurements became loose during the test resulting in extremely erratic results. The data in Figures 16-19 are also presented in Figures 20 through 23 with shrinkage plotted against the log of the water content.

The shrinkage and capacitance reading at approximately the same water content are compared for different phases of wetting and drying on Figures 24 through 26.

The results of the low frequency tests show that at the frequency of 60 cycles per second the method is very sensitive to changes in water content at low water contents (below about 2%) but very insensitive at higher water contents. Comparing this range of sensitivity to the range over which most of the volume change occurs it is seen that about 40% of the volume change takes place in the range where the capacitance measurements are insensitive (see Figures 24 and 25). Significant volume changes accompany moisture changes up to water contents of approximately 5 or 6% after the first drying. This is somewhat higher than the values indicated in the literature for concrete. In concrete, the presence of the relatively inert coarse aggregate moves the volume change-water content curve to the left. The corresponding

FIG. 16- SHRINKAGE vs. WATER CONTENT - DISK # 9

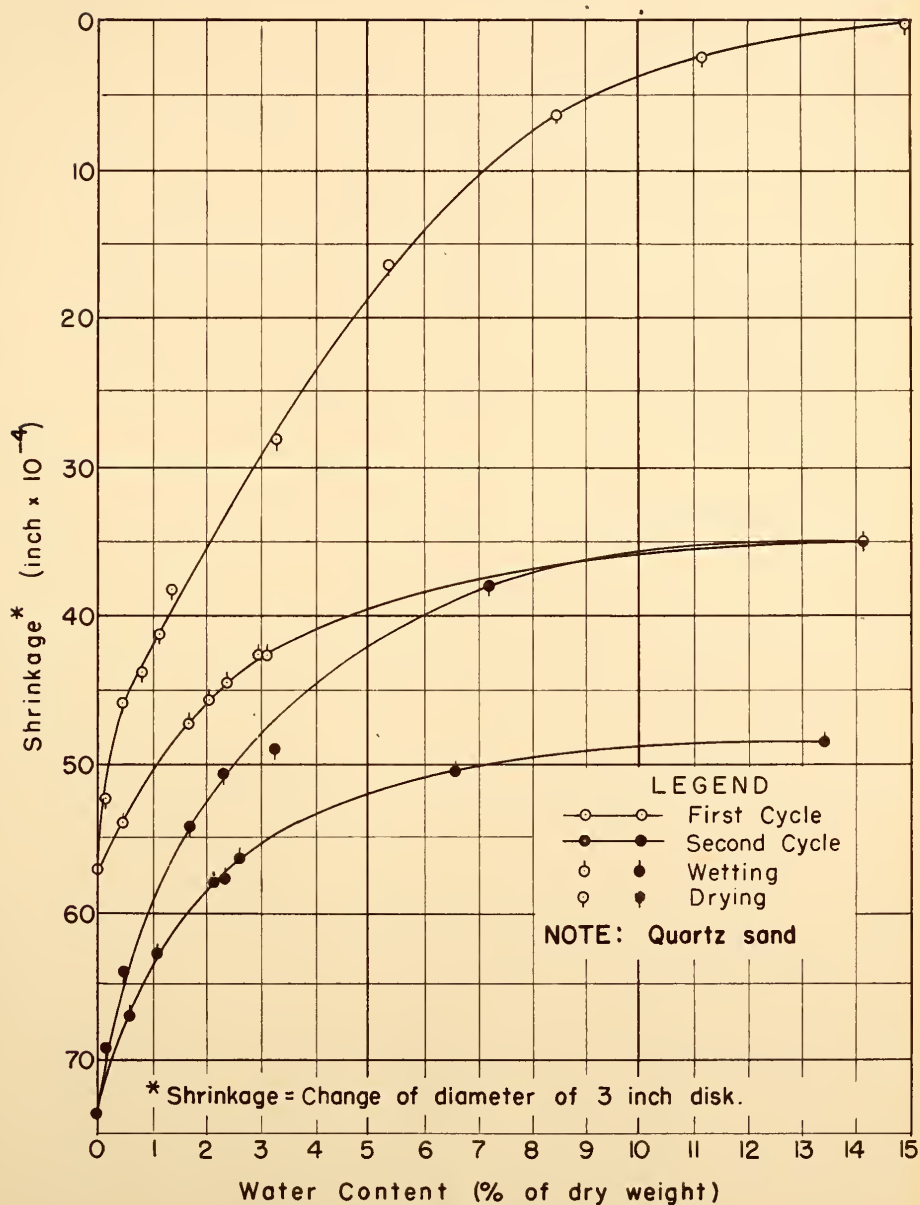


FIG. 17-SHRINKAGE vs. WATER CONTENT- DISK # 2

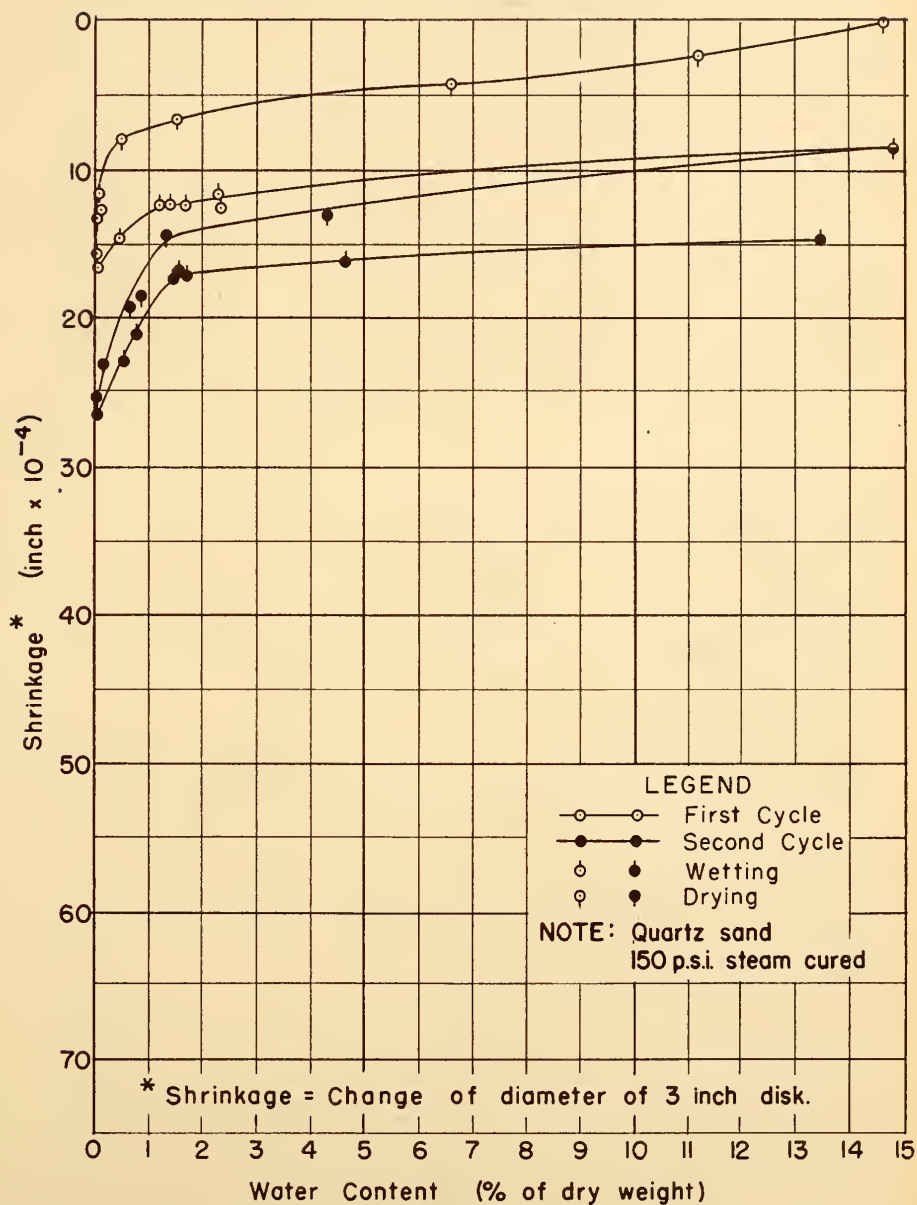


FIG. 18 - SHRINKAGE Vs WATER CONTENT- DISK C-S

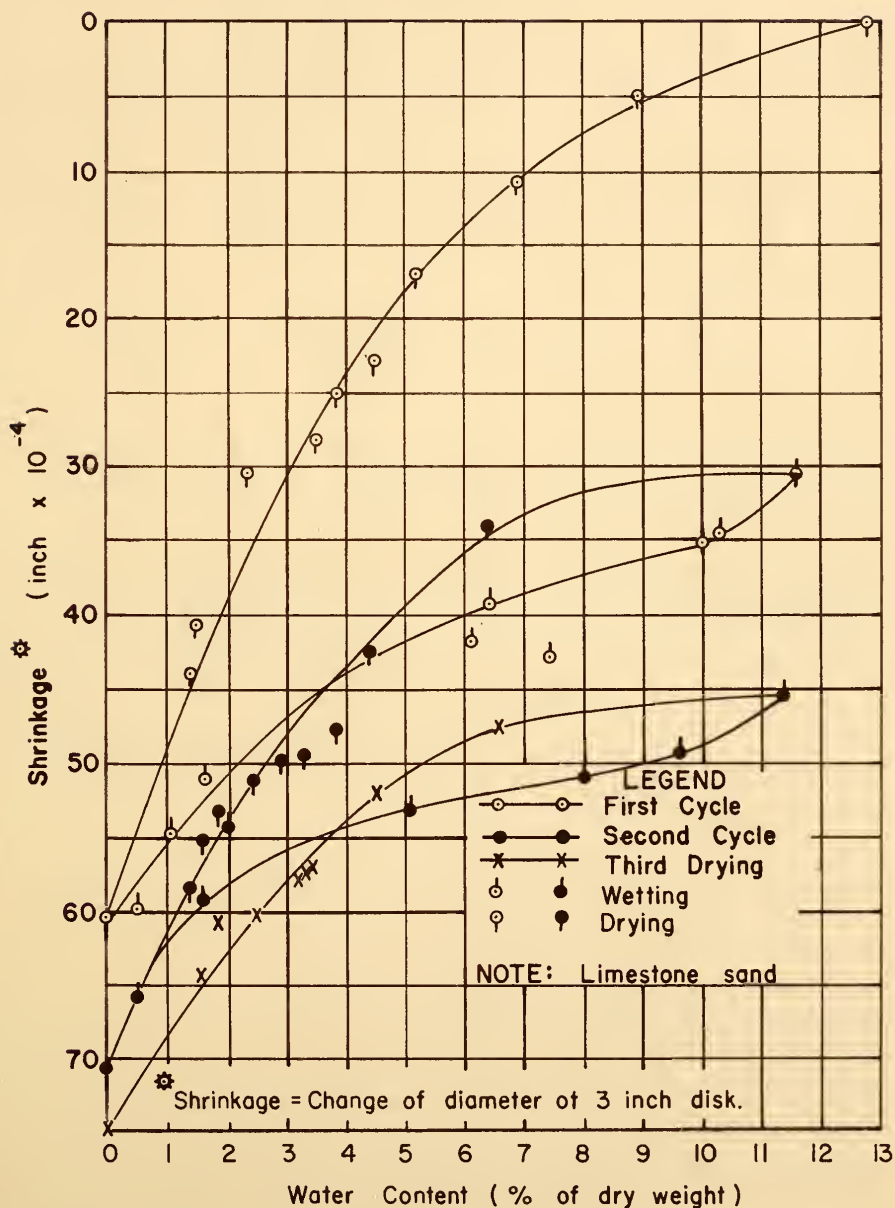


FIG. 19 - SHRINKAGE Vs WATER CONTENT- DISK B-S

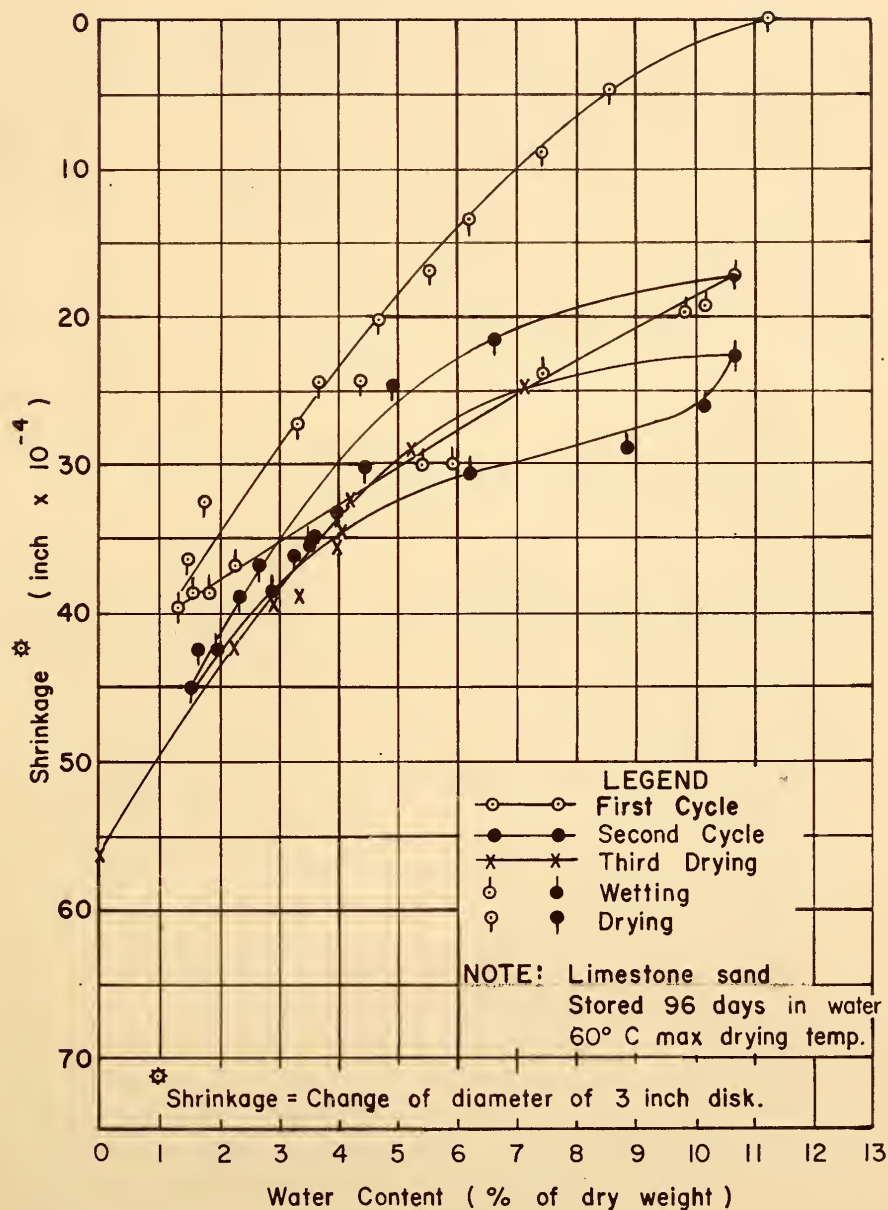


FIG. 20 - SHRINKAGE VS LOG OF WATER CONTENT - DISK 9

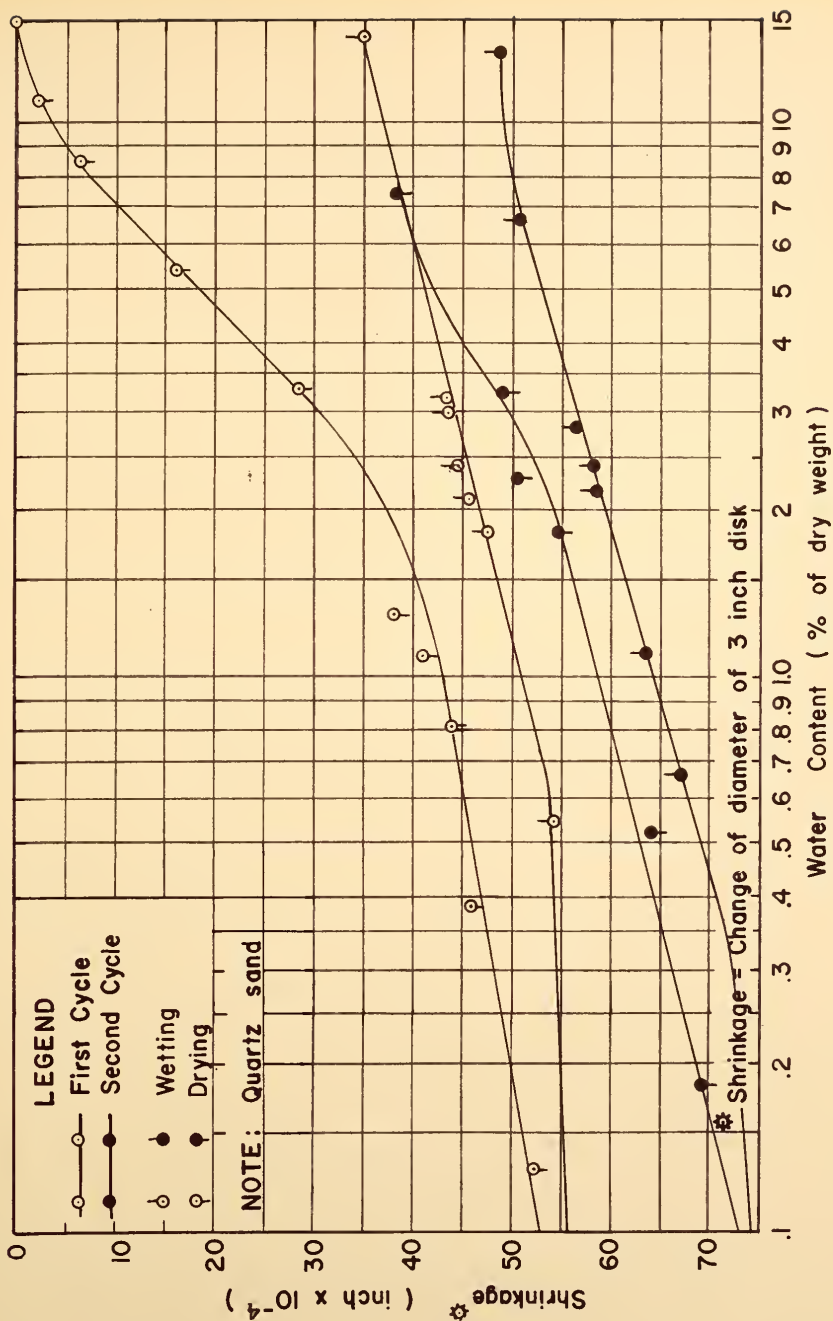


FIG. 21 - SHRINKAGE Vs LOG OF WATER CONTENT - DISK 2

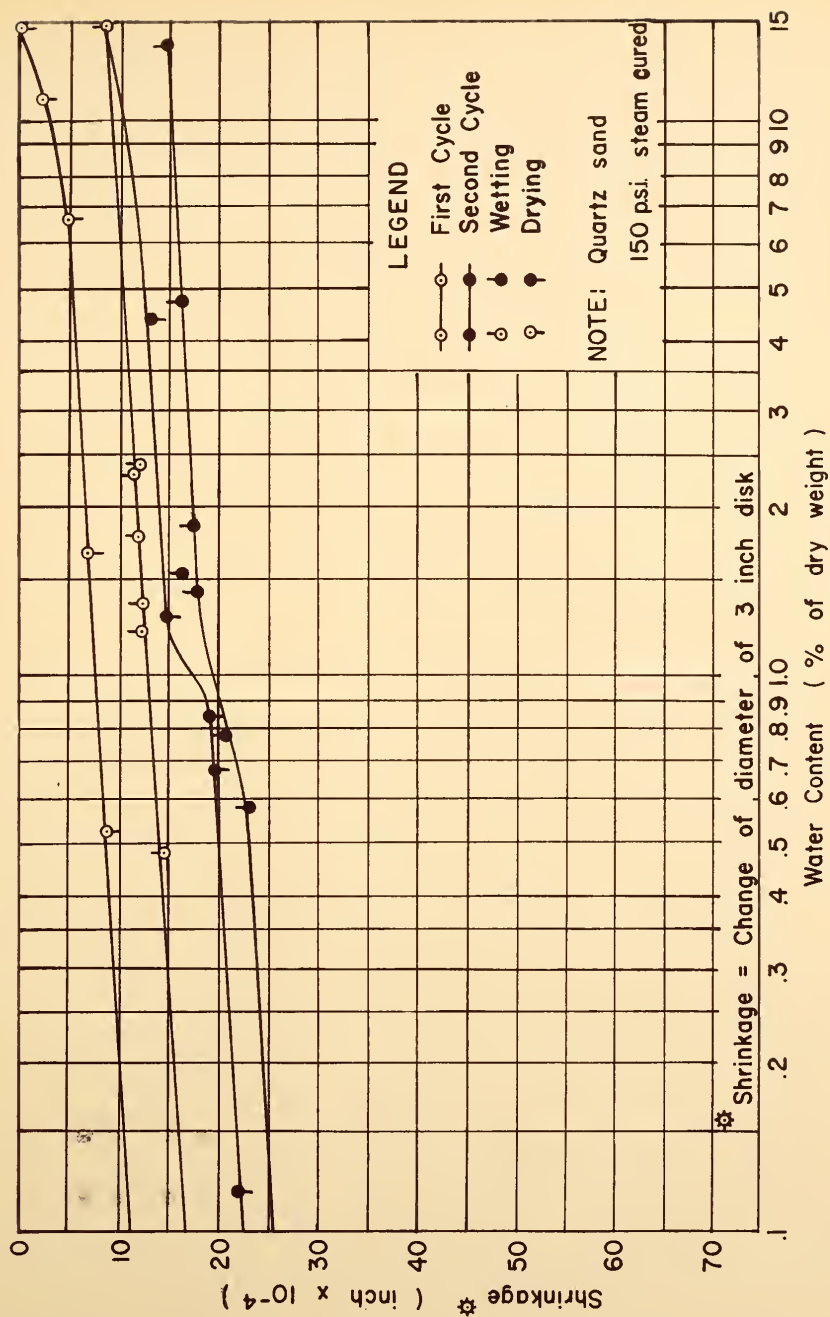


FIG. 22 - SHRINKAGE Vs LOG OF WATER CONTENT - DISK C-S

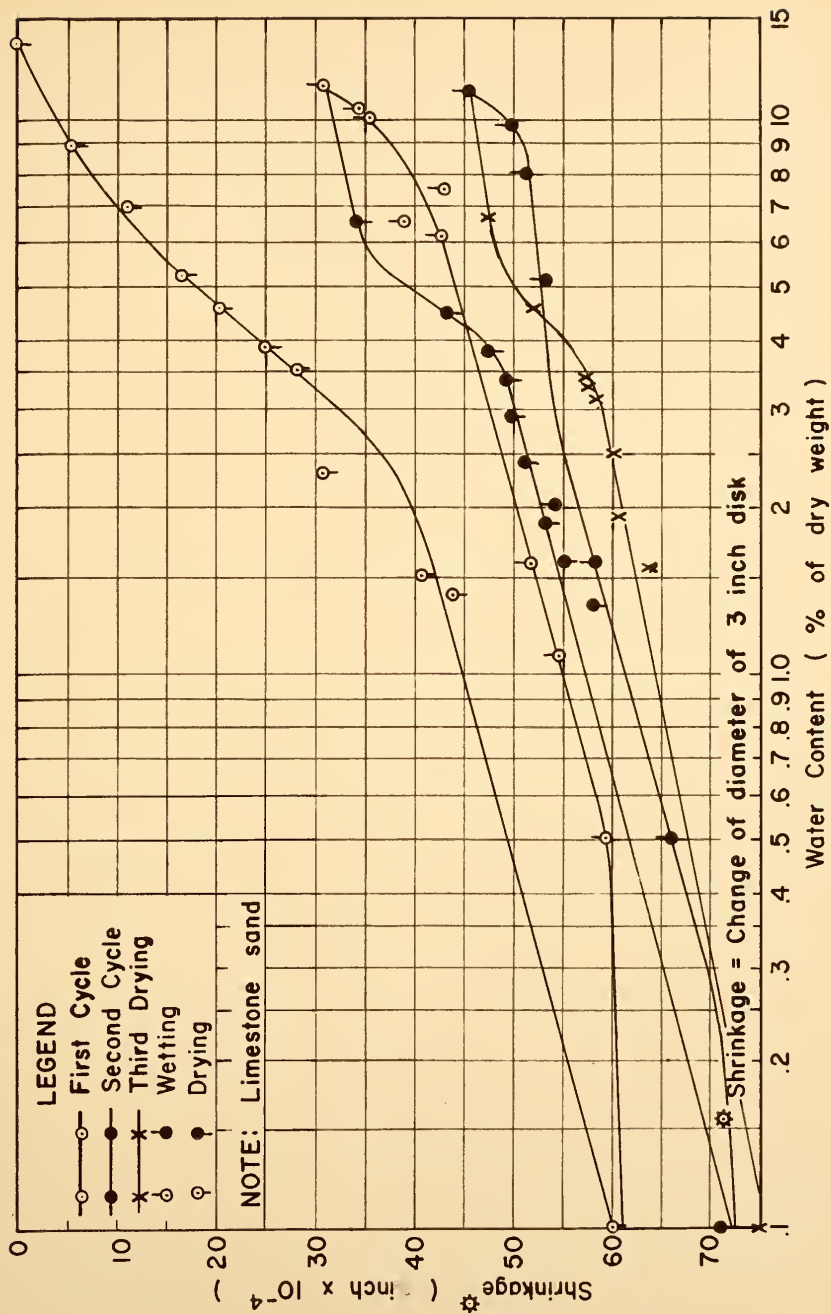


FIG. 23 - SHRINKAGE Vs LOG OF WATER CONTENT- DISK B-S

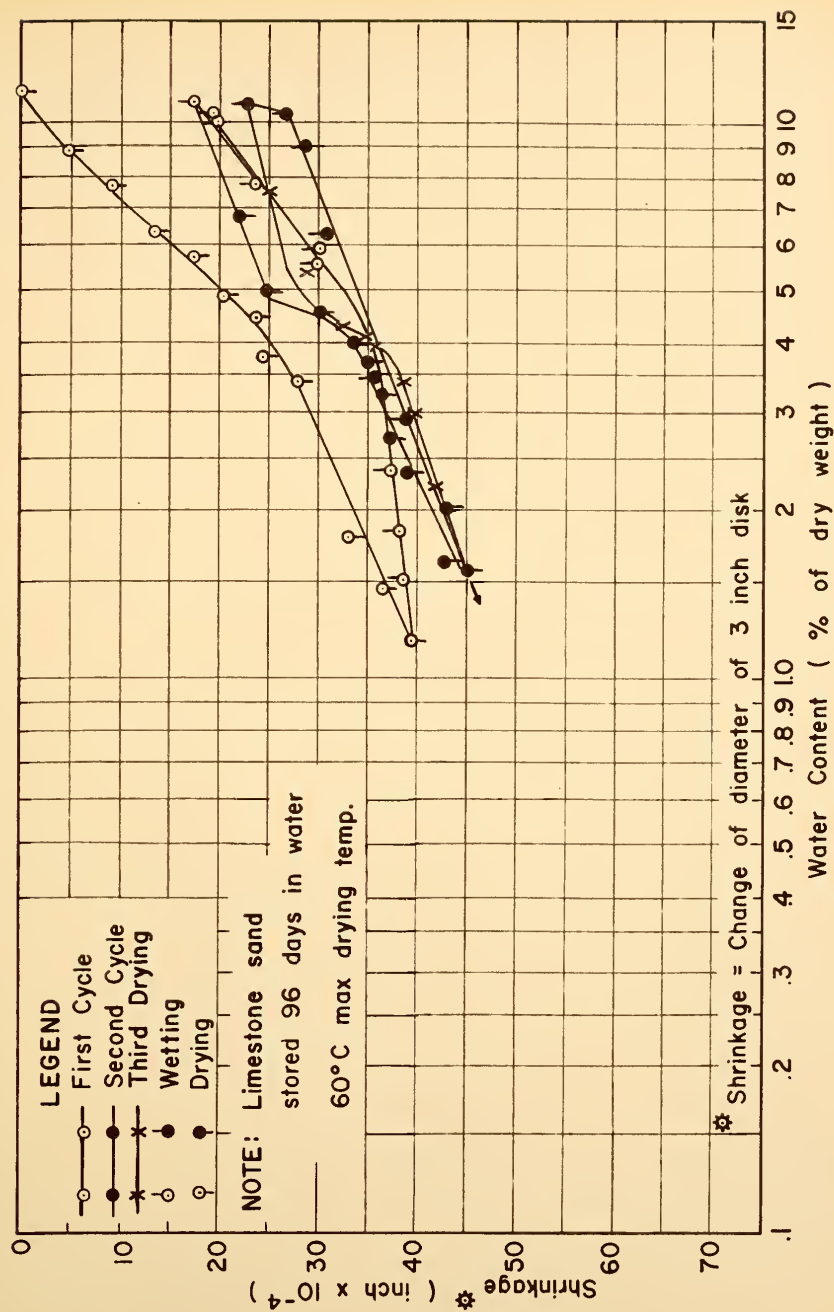


FIG.24- CAPACITANCE vs. PERCENT OF SHRINKAGE
PER WETTING OR DRYING PHASE.-DISKS # 8 & 9.
(60 c.p.s.)

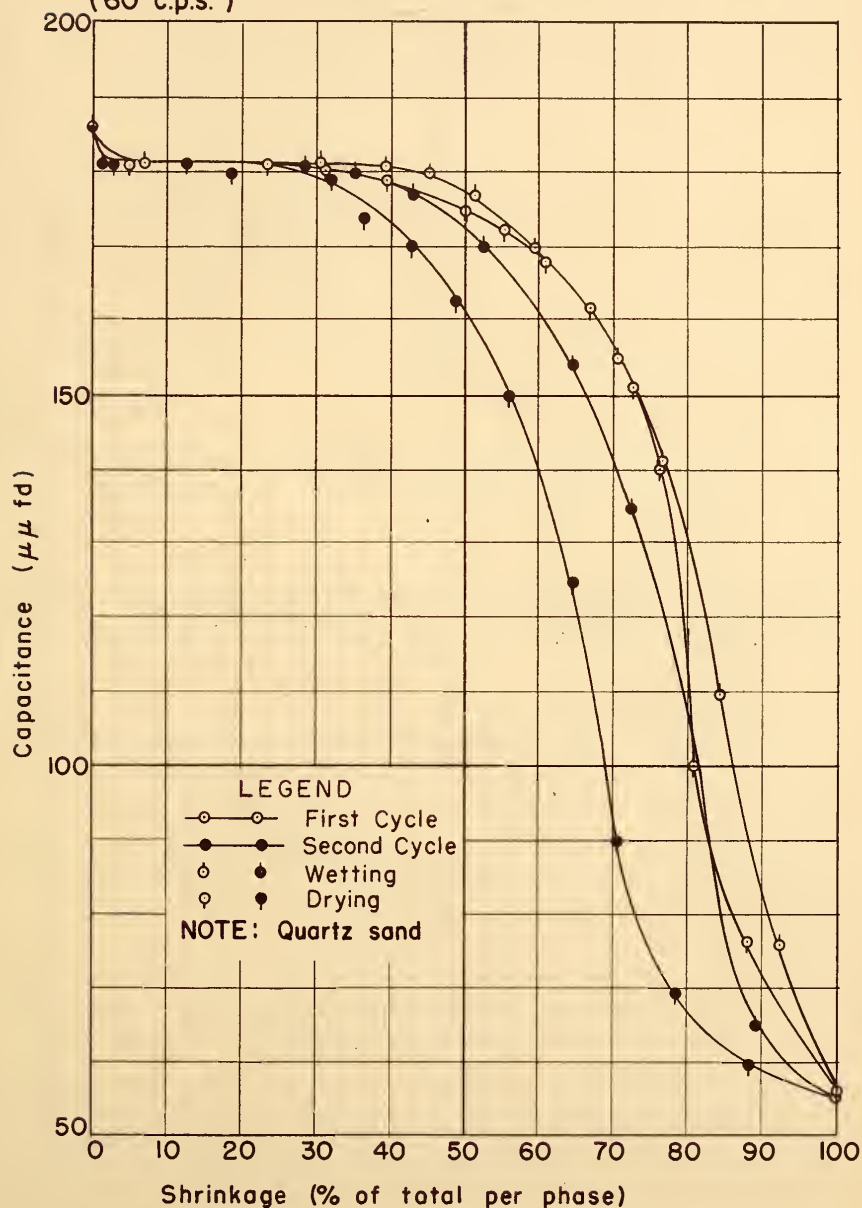


FIG.25- CAPACITANCE vs. PERCENT OF SHRINKAGE
PER WETTING OR DRYING PHASE.- DISKS #1 & 2.
(60 c.p.s.)

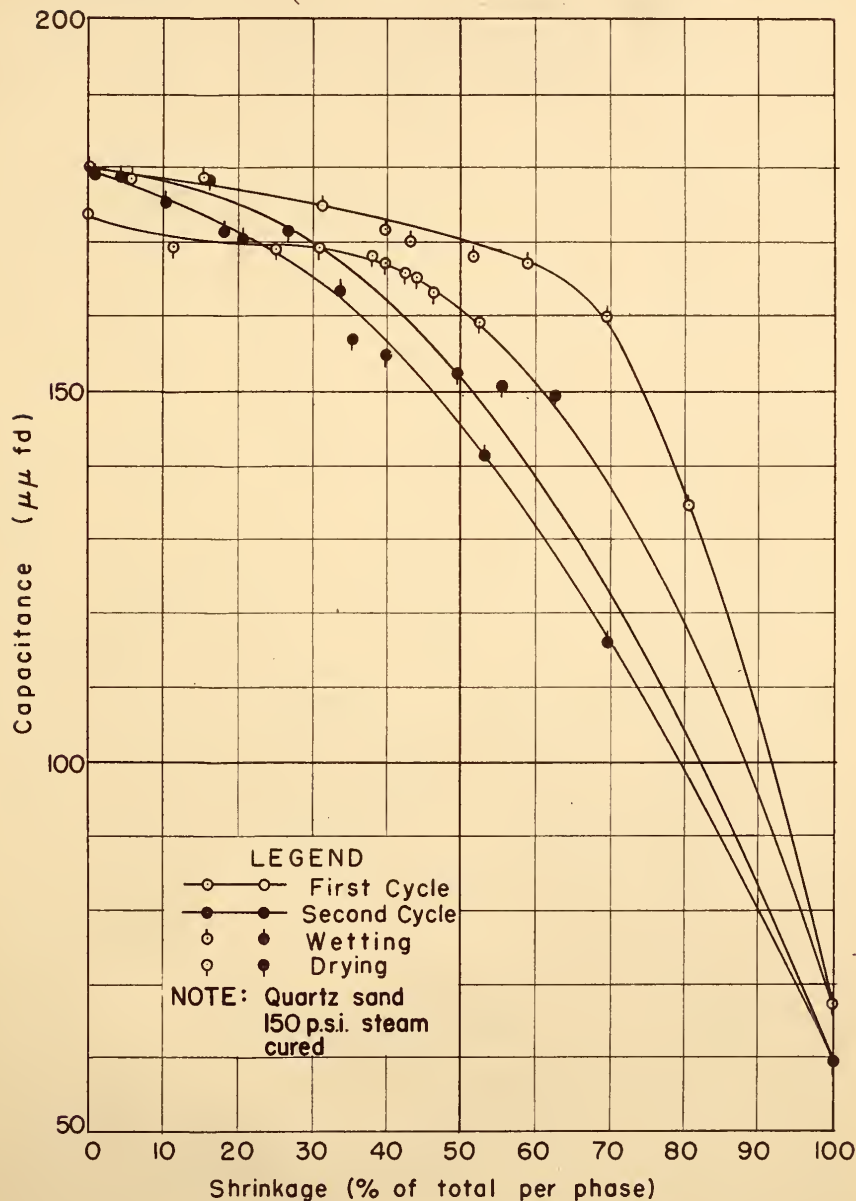
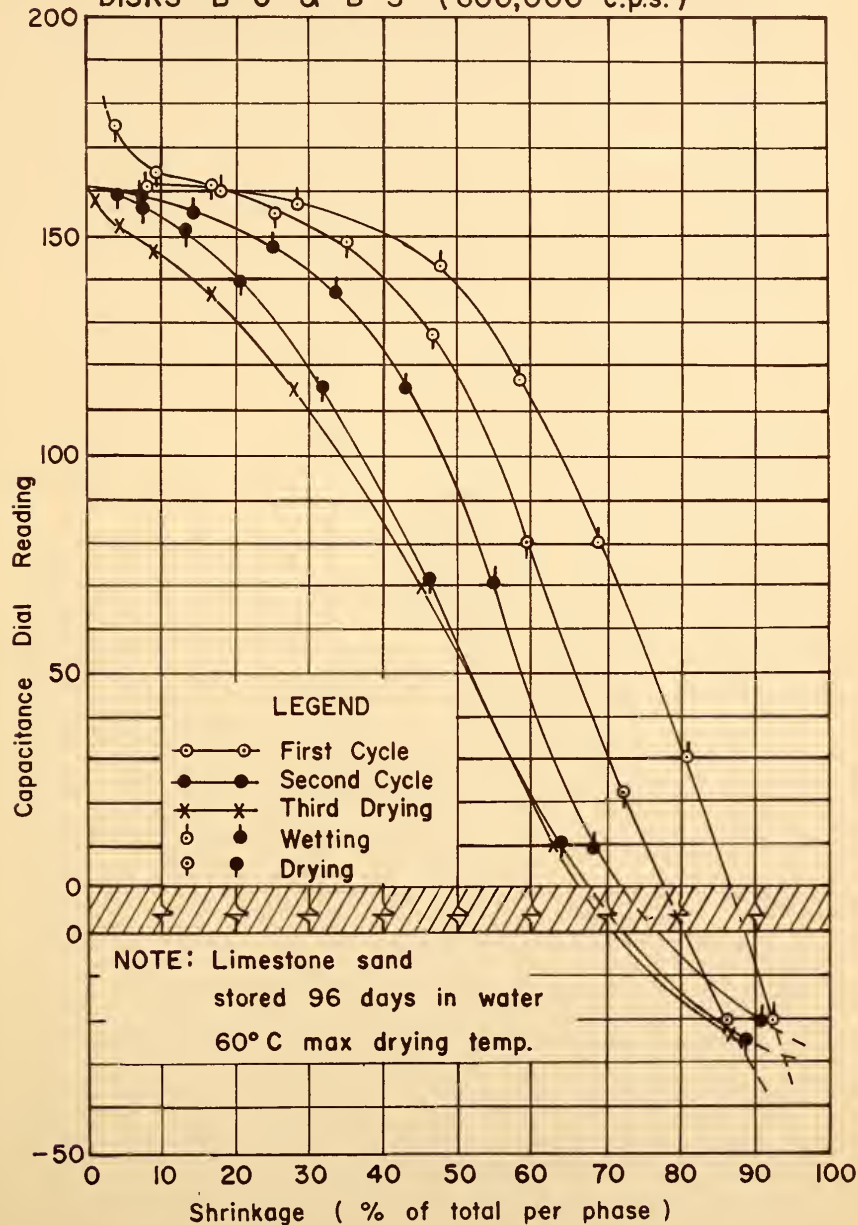


FIG. 26 - CAPACITANCE DIAL READING Vs PERCENT OF SHRINKAGE PER WETTING OR DRYING PHASE DISKS B-C & B-S (600,000 c.p.s.)



capacitance - water content curve would also probably be moved to the left and the two curves would maintain approximately the same relative positions as those obtained in the tests on mortar. However, the results of the high frequency tests (600,000 cycles per second) show that at this frequency the method is sensitive to changes in water content over the full range significant with respect to shrinkage (see Figure 22).

The capacitance curves show a hysteresis loop in the wetting-drying cycle with the re-wetting curve shifted slightly to the left at low water contents. This, possibly, results from the same factors which cause the hysteresis in the sorption curves for concrete (54). It is interesting to note that hysteresis was almost eliminated when the disk was dried at 60°C instead of 105°C.

The capacitance curves also show a shift to the right on the second and third drying cycles. This shift was not indicated by the tests on Disk #8 in the high frequency tests except at high water contents. As this disk had been through several cycles of wetting and drying before the start of the tests, it is probable that this shift continues for a large number of cycles but the magnitude of the shift becomes small after the first few cycles. The magnitude of this shift was also greatly reduced when the mortar was dried at a temperature of 60°C rather than 105°C. The shrinkage - water content curves also demonstrate a similar shift on successive cycles.

The capacitance curves for all disks are of the same general shape but the absolute values vary appreciably from disk to disk. This results from the different structures of the mortars, as the density and the dielectric constant of the solids are approximately constant from sample to sample. These differences may also reflect variations in

the quality of the contact between the disks and the plates of the test capacitor. The influence of the contact capacitance will be greatest at high water content. In the high frequency tests, when the structures of different disks are similar their capacitance - water content values are nearly the same except at high water contents.

DISCUSSION OF RESULTS

The results of the low frequency tests show that at very low electrical frequencies the capacitance method is not an adequate indicator of water content because it is not sensitive over the full range of moisture content that is of interest. However, the high frequency tests show that this deficiency can be overcome without reducing the sensitivity below an acceptable value. At 600,000 c.p.s. the method is sensitive up to water contents in excess of 6%.

The differences between the results at the different frequencies result from the fact that the apparent dielectric constant as measured by capacitance is different from the true dielectric constant of the material. First, at the low frequencies used, polarization films may have developed around the capacitor plates causing apparent dielectric constants higher than the true value for distilled water. Smith - Rose (53) has shown that this effect may be significant at frequencies up to the low R. F. frequencies, which are much higher than the frequencies normally employed to eliminate polarization effects in resistivity measurements. At high frequencies this polarization does not occur and the dielectric constant of salt water is very nearly equal to the value for distilled water. Second, changes in conductivity of the dielectric are reflected in the values of the measured apparent dielectric constant. Childs (55) demonstrates that at low frequencies this effect may be large but at high frequencies it is negligible. The data by Blyth (22) and Spencer (17) show that very large changes in the conductivity of concrete accompany small changes in water content below

about 3 percent. This is the approximate range over which most of the capacitance change occurred in the low frequency phase of this study. Increasing the frequency, therefore, not only increases the range of the method but it also reduces the effect of changes in the salt concentration of the pore fluid. The belief that at high frequencies the method is not sensitive to changes in the composition of the pore water is substantiated by the data for Disk O (Figure 14). After two wetting-drying cycles the dried disk was saturated with a 1/2 normal calcium chloride solution and capacitance was measured through a third cycle. The results of this test show that, within the accuracy of the measurements, the increased salt concentration of the pore water had no noticeable effect.

Capacitance - water content curves obtained by other investigators show the same trends as those from this study. Curves obtained by Anderson (35) for soils at low frequencies are very similar in shape to the curves obtained in this study but curves obtained for soils at higher frequencies by Edlofsen (37) and Balls (42), and for gelatin and paper pulp by Besser and Piret (43) exhibit more nearly linear relationships. The data for gelatin and paper pulp were obtained at several frequencies in the low mega-cycle range. These curves become progressively flatter as the frequency is increased. In addition, companion conductivity determinations indicate that at these frequencies the apparent dielectric constant is essentially free of the influence of conductivity.

The range of the method could be extended even farther by increasing the frequency above the 600,000 c.p.s. used in the high frequency series in this study. This frequency is not very high; it corresponds approximately to the lowest commercial radio frequencies. The common television

frequencies are several hundred times as high; therefore, it would not require any special or difficult techniques to make capacitance measurements at frequencies considerably higher than those used in this investigation.

The most serious limitations to the dielectric constant as a moisture indicator brought out by this study are the hysteresis in the capacitance - water content curves and the continued shifting of the curves to the right as the structure of the concrete changed with time. In addition, this latter factor suggests that different samples of concrete from the same batch might even have different capacitance - water content relationships as a result of differences in placing and curing. There are, however, certain indications in the results that these limitations may not be as serious as they first appear.

Figure 13 shows the results of the 600,000 c.p.s. tests on Disk #8. This disk had been through several cycles of wetting and drying and had been thoroughly air dried before the tests (see Table 2) and, therefore, should have been relatively stable with respect to structural changes during the test period. These curves do not show the shift to the right on subsequent cycles, and the differences between cycles appears to be completely random. This disk also exhibits a smaller hysteresis than the fresher disks. This smaller hysteresis may, however, be a result of the quartz aggregate rather than more complete curing because the low frequency tests on quartz sand mortars also show less hysteresis. As low frequency tests were not performed on limestone sand mortars it is not possible to establish whether the reduced hysteresis is due to the frequency or to the aggregate.

The tendency for the curve shifting to decrease rapidly after only a few cycles is also shown for Disk A-C (Figure 10). The second drying curve for this disk is quite different than the first but the third is almost identical to the second. Disk C-C (Figure 11) demonstrates this same phenomenon but to a lesser degree. The third drying curve still deviates somewhat from the second but this disk had only been stored one day before testing whereas Disk A-C had been kept in water 96 days before testing. Both of these disks show variations in water content of about 1% due to hysteresis at low water contents.

On Figure 15 it is shown that after $1\frac{1}{2}$ drying - wetting cycles the capacitance - water content relationships for Disks A-C and C-C are the same. This is interesting because initially the two disks were not the same and had different capacitance - water content curves on the first cycle. The disks were prepared from mortars of the same mix design but from different batches and Disk A-C was kept in water 96 days to only one day for Disk C-C. The resulting difference in structure of the mortar is reflected by the data on Figure 6. These data show that for the first drying phase the sorption relationships for the two disks were quite different; however, after the second drying the two curves were very similar indicating essentially the same structure. This is substantiated by the capacitance data.

Possibly the most significant data obtained in this investigation are presented as Figure 12. This figure shows the results of 600,000 c.p.s. capacitance tests on Disk B-C. This disk was dried at a maximum temperature of 60°C instead of the standard 105°C . Disk B-C was prepared from

the same cylinder as Disk A-C and in all respects was treated the same as A-C before testing. The fact that these two disks were initially very similar is shown by the similarity of the first desorption curves on Figure 6. But after the first drying, when Disk A-C was dried at 105°C while Disk B-C was dried at only 60°C , the sorption curves indicate a definite difference between the structures of the disks. The first drying phase capacitance - water content curves for these two disks are similar and below water contents of 5% are nearly identical. After the first drying the curves for the two disks are markedly different. Except at very high water contents the capacitance - water content curve for Disk B-C does not show any significant shifting and shows very little if any hysteresis. For water contents below about 5% all data points - all cycles and wetting and drying - can be represented very well by one curve. This suggests that the hysteresis effect may be a serious limitation only if the concrete is dried at a relatively very high temperature. The capacitance curves for the first drying phase of Disks B-C and C-C do not coincide as they did for Disks B-C and A-C. The curve for C-C is off-set approximately $1/2\%$ to the left. This disk was not as well cured as Disks B-C and A-C; therefore, it is probable that there will be a slight shifting of the capacitance curves to the right for the first several cycles even though such shifting is not pronounced for Disk B-C. This will be especially true for concretes which are not well cured.

Although the number of tests made in this investigation was small, there is strong evidence that the dielectric constant is a reliable indicator of the evaporable moisture content of hardened concrete, at least

after the first few wetting - drying cycles particularly if the concrete is not heated to exceptionally high temperatures.

At 600,000 c.p.s. the method is relatively insensitive and somewhat erratic at high water contents above 6%. The range can be extended farther by increasing the frequency. Casting a sensing element in the concrete, as would be done in a field test, will produce a better contact than is possible by simply clamping a disk in the test capacitor and will also improve the sensitivity and reliability at high water contents.

The shrinkage tests performed in connection with this study had two purposes. The first was to establish the range of moisture contents over which most of the shrinkage occurred. The second was to determine approximately the moisture coefficient of expansion. These factors are functions of many variables such as curing, cement, aggregate, and moisture history, which were not thoroughly investigated in this study. However, the requirements for a satisfactory meter to appraise the effects of moisture gradients on warping can be estimated from the data available.

Examination of Figures 16 through 19 shows that by far the greatest rates of volume change occur at water contents less than about 7%. The first drying may be an exception to this. This is somewhat higher than the values indicated in the literature for concrete. In concrete, the presence of the relatively inert coarse aggregate moves the volume change - water content curve to the left, reducing the range requirement. For the high frequency tests the range of the capacitance method is adequate to cover this range. Figure 26 shows that at 600,000 c.p.s. only about 10 - 15% of the shrinkage occurred outside the range of sensitivity of the method after the first cycle.

Neglecting the first drying, the maximum value for the moisture coefficient of expansion of mortar indicated in this study is approximately 5×10^{-4} inches per inch per percent moisture content for Disk #9 at water contents below 1%. This corresponds approximately to the effect of a temperature variation of 50°F . These facts, combined with the data in the literature, show that if moisture contents are to be used to predict volume changes with an accuracy equivalent to the volume change resulting from a temperature change of 5°F , the moisture determination must be accurate to within $\pm 0.1\%$. At the present state of knowledge such accuracy could not be achieved by indirect determinations.

For practical problems such precise measurements would not be required. In a highway pavement slab there would be three factors acting to reduce the accuracy required. First, the tests on Disks B-C and B-S which were dried at 60°C (140°F) show that the minimum water content would always be appreciably above that corresponding to the range of maximum rate of shrinkage, and the moisture coefficient of expansion would therefore be considerably smaller than the maximum given above. For Disk B-S, the coefficient of expansion at a water content of 2% is only about $1/3$ the value given above. Second, with each drying of the concrete the rate of shrinkage decreases. Third, the large amount of relatively inert coarse aggregate in concrete acts to reduce the moisture coefficient of expansion of concrete relative to that of mortar. Shideler's data (8) indicates values for concrete about $1/2$ that found for the mortar used in this study. These factors will reduce the effective moisture coefficient of expansion so that an accuracy in moisture measurement of $\pm \frac{1}{2}\%$ would probably be satisfactory for most practical field problems.

The shrinkage data show that, in using moisture content as an indicator of volume change, measuring the moisture content is only half the problem. The other half is to predict the shrinkage after the water content has been determined. The problem arises from the fact that the volume - water content relationship of a concrete is constantly changing. Therefore, it must be possible to predict how this relationship will change with time.

The measurements made in this study were not sufficiently precise or comprehensive to provide a basis for such predictions, but they do show certain trends. The semi-log plots of the shrinkage data (Figures 20 through 23) are particularly interesting. The values of shrinkage show considerable variation between disks, but the curves are all similar in shape and undergo the same evolution. The regularity of the changes in the shrinkage - moisture relationship upon successive wetting - drying cycles suggest that these changes may be predictable; however, this problem requires much more study.

CONCLUSIONS AND RECOMMENDATIONS

On the basis of the information obtained in this study, it is concluded:

1. To be satisfactory for use in connection with moisture - volume relationship investigations, a moisture meter must be capable of measuring water contents in hardened concrete between about 1 and 4 or 5 percent. For mortars the range of interest extends to 6 or 8 percent.
2. To indicate volume changes due to moisture variations with an accuracy which will limit the error to an amount less than the volume change due to a temperature change of 5°F, the accuracy of the moisture measurements must be at least $\pm\frac{1}{2}$ of one percent for most cases. At very low water contents and for the first drying this accuracy may not be adequate.
3. On the basis of the known inherent limitations and advantages, the dielectric constant or capacitance moisture meter appears the most promising. The ionic barrier moisture meter is also promising and, in the event that additional experimentation should prove the capacitance method impractical, is deserving of additional study.
4. For low electrical frequencies the capacitance method is very sensitive at very low water contents but is generally unsatisfactory because of its extremely narrow range of sensitivity; however, at higher frequencies the range of sensitivity is extended to include the higher water contents. It might prove beneficial to use different frequencies for different moisture ranges.

5. The capacitance moisture measuring method is sensitive to changes in the structure of the concrete with time and reflects the hysteresis in the sorption relationship of the concrete. These are factors which also limit the accuracy of all other known indirect methods of measuring evaporable water content. For the capacitance method it appears likely that the concrete becomes sufficiently stable after only a few cycles of wetting and drying to eliminate this as a serious problem. Also, the hysteresis does not cause errors in excess of 1/2% except possibly at very low water contents and for samples which have been dried at temperatures in excess of 60°C.
6. In studies where water content is to be used to predict the shrinkage of concrete, the problem of establishing the water content - volume relationship at any given time is equally as important as the actual moisture measurement because this relationship is constantly changing, and among other things, is a function of the moisture history of the concrete which would be unique for each installation. The results of the shrinkage tests conducted in conjunction with this study show some consistent trends which indicate that it may be possible to predict the moisture - volume relationship at any time if the moisture history is fairly well known. If this is not possible, it may at least be possible to predict relative volume changes, that is, to predict that a given moisture change would cause a certain volume change even though the absolute shrinkage relative to the original volume might not be known. This problem must be given additional study before

the capacitance meter (or any other moisture meter) can be used to indicate the volume changes associated with moisture changes in hardened concrete.

In the light of the above conclusions, the following additional studies are recommended.

1. An intensive research program should be initiated to develop the capacitance moisture measuring method into a practical research tool. This program would have two parts. The first phase would consist of additional studies of the capacitance - water content relationship. These studies would be designed to investigate the effects of aggregate type and quantity, mix design, curing, and electrical frequency of measurement in sufficient detail to provide a basis for the final design of the moisture meter and for the interpretation of insitu measurements. The second phase of the program would be devoted to actually developing the instruments and procedures for insitu moisture measurements of hardened concrete.

The instrumentation phase should be planned to develop all equipment and procedures necessary to permit moisture measurements on a routine basis. In addition to the actual capacitance bridge this would include the sensing element which would be embedded in the concrete, all leads and connections, installation procedures, calibration methods, and temperature corrections.

2. A concomitant investigation of the moisture - volume relationships of concrete should be conducted. The purpose of this study would

be to make it possible to predict shrinkage from water content at any time if the moisture history of the concrete were known.

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